

# **STIC Search Report**

## **Biotech-Chem Library**

STIC Database Tracking Number: 115545

**TO:** Shailendra Kumar  
**Location:** Rem 5d61 / 5c18  
**Monday, March 08, 2004**  
**Art Unit:** 1621  
**Phone:** 272-0640  
**Serial Number:** 09 / 774232

**From:** Jan Delaval  
**Location:** Biotech-Chem Library  
Rem 1A51  
**Phone:** 272-2504  
  
**[jan.delaval@uspto.gov](mailto:jan.delaval@uspto.gov)**

### Search Notes

Jan Please

14902

Access DB# 115545

## SEARCH REQUEST FORM

### Scientific and Technical Information Center

Requester's Full Name: S.Kumar Examiner #: 69594 Date: 2/23/04  
Art Unit: 162 Phone Number 305-772-0610 Serial Number: 09/774,232  
Mail Box and Bldg/Room Location: EM 5D61 Results Format Preferred (circle): PAPER DISK E-MAIL  
5C70

If more than one search is submitted, please prioritize searches in order of need.

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc., if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Material Processing by Repeated Solvent Expansion-contraction

Inventors (please provide full names): Said Sawim et al.

Earliest Priority Filing Date: 3/3/2000

\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.  
(The solute is acetanetophen)

A method for producing solute comprising

- a) dissolving solute in a liquid solvent to form solvent/solute phase
- b) dissolving gaseous fluid in the solvent/solute liquid phase
- c) causing solvent/solute/gaseous liquid phase to expand
- d) causing gaseous fluid in the dissolution to a concentration such that the solvent/solute/gaseous fluid liquid phase expands until it loses its affinity for solubilization of said solute and said solute precipitates
- e) retaining precipitated solute on a retention medium
- f) reducing the pressure in the liquid phase to a point where a
- g) optionally adding more solute to the liquid phase.

See claims 1-11 + 12-16

STAFF USE ONLY		Type of Search	Vendors and cost where applicable
Searcher:	<u>Jam</u>	NA Sequence (#)	STN
Searcher Phone #:	<u>72504</u>	AA Sequence (#)	Dialog
Searcher Location:		Structure (#)	Questel/Orbit
Date Searcher Picked Up:	<u>3/8</u>	Bibliographic	Dr. Link <u>101191</u>
Date Completed:	<u>3/8</u>	Litigation	Lexis/Nexis <u>101823</u>
Searcher Prep & Review Time:		Fulltext	Sequence Systems <u>101823</u>
Clerical Prep Time:	<u>1</u>	Patent Family	WWW/Internet <u>101823</u>
Online Time:	<u>+ 100</u>	Other	Other (specify) _____

=> fil reg

FILE 'REGISTRY' ENTERED AT 14:51:01 ON 08 MAR 2004  
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.  
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.  
COPYRIGHT (C) 2004 American Chemical Society (ACS)

Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 7 MAR 2004 HIGHEST RN 659718-58-8  
DICTIONARY FILE UPDATES: 7 MAR 2004 HIGHEST RN 659718-58-8

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2004

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:  
<http://www.cas.org/ONLINE/DBSS/registryss.html>

=> d 11 ide can

L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN  
RN 124-38-9 REGISTRY

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

OTHER NAMES:

CN Carbon oxide (CO<sub>2</sub>)

CN Carbon-12 dioxide

CN Carbon-12C dioxide-16O<sub>2</sub>

CN Carbonic acid anhydride

CN Carbonic acid gas

CN Carbonic anhydride

CN Dry ice

CN Khiadon 744

CN R 744

FS 3D CONCORD

DR 18923-20-1

MF C O<sub>2</sub>

CI COM

LC STN Files: ADISNEWS, AGRICOLA, ANABSTR, AQUIRE, BIOBUSINESS, BIOSIS, BIOTECHNO, CA, CABA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CBNB, CEN, CHEMCATS, CHEMINFORMRX, CHEMLIST, CHEMSAFE, CIN, CSCHEM, CSNB, DDFU, DETHERM\*, DIOGENES, DIPPR\*, DRUGU, EMBASE, ENCOMPLIT, ENCOMPLIT2, ENCOMPPAT, ENCOMPPAT2, GMELIN\*, HODOC\*, HSDB\*, IFICDB, IFIPAT, IFIUDB, IPA, MEDLINE, MRCK\*, MSDS-OHS, NIOSHTIC, PDLCOM\*, PIRA, PROMT, RTECS\*, SPECINFO, TOXCENTER, TULSA, ULIDAT, USAN, USPAT2, USPATFULL, VETU, VTB  
(\*File contains numerically searchable property data)

Other Sources: DSL\*\*, EINECS\*\*, TSCA\*\*

(\*\*Enter CHEMLIST File for up-to-date regulatory information)

O=C=O

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

174398 REFERENCES IN FILE CA (1907 TO DATE)

669 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA

174609 REFERENCES IN FILE CAPLUS (1907 TO DATE)  
 21 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:173564

REFERENCE 2: 140:173485

REFERENCE 3: 140:173471

REFERENCE 4: 140:173329

REFERENCE 5: 140:173266

REFERENCE 6: 140:173203

REFERENCE 7: 140:172374

REFERENCE 8: 140:172051

REFERENCE 9: 140:172028

REFERENCE 10: 140:172024

=> d his

(FILE 'HOME' ENTERED AT 13:39:27 ON 08 MAR 2004)  
 SET COST OFF

FILE 'REGISTRY' ENTERED AT 13:39:36 ON 08 MAR 2004

L1 1 S CARBON DIOXIDE/CN

FILE 'HCAPLUS' ENTERED AT 13:39:44 ON 08 MAR 2004

L2 174782 S L1  
 L3 438211 S CO2 OR CARBON() (DIOXIDE OR DI OXIDE)  
 L4 443621 S L2,L3  
     E SOLUTE/CT  
 L5 4846 S E20-E23  
     E E20+ALL  
 L6 4846 S E2  
 L7 1962 S DISSOLVED SUBSTANCE  
 L8 78132 S E4-E27  
 L9 6713 S L5-L7  
 L10 1980 S SOLVENT AND L9  
     E SOLVENT/CT  
 L11 46150 S E53-E85  
     E E53+ALL  
 L12 48190 S E2+NT  
     E E18+ALL  
 L13 45384 S E2,E1+NT  
 L14 1062 S L9 AND L11-L13  
 L15 1133 S L8 AND L11-L13  
 L16 7863 S L8 AND SOLVENT  
 L17 9795 S L10,L14-L16  
 L18 512 S L17 AND (GAS OR GASEOUS)  
     E GAS/CT  
 L19 0 S L17 AND E3  
     E GASES/CT  
 L20 131 S L17 AND E3+NT  
 L21 20 S L17 AND E3-E25  
     E E3+ALL  
 L22 0 S L17 AND E5

L23 188 S L17 AND L4  
 L24 703 S L18, L20, L21, L23  
 L25 25 S L24 AND (EXPAND? OR EXPANSION?)  
 L26 0 S L24 AND RETRACT?  
 L27 3 S L24 AND CONTRACT?  
 L28 22 S L24 AND PRECIPITAT?  
 L29 48 S L24 AND EXTRACT?  
 L30 36 S L24 AND COAT?  
 L31 23 S L24 AND RETENTION  
 L32 11 S L24 AND ?FILTR?  
 L33 16 S L24 AND ?FILTER?  
 L34 34 S L24 AND ?CRYST?  
 L35 170 S L25-L34  
 L36 8 S L35 AND LIQUID PHASE  
     E SAIM S/AU  
 L37 27 S E3, E4  
     E HORHOTA S/AU  
 L38 15 S E4-E8  
     E BOCHNIAK D/AU  
 L39 5 S E4-E6  
     E BOEHRING/PA, CS  
 L40 8232 S E4-E9 OR BOEHRINGER?/PA, CS  
     E BOHRINGER/PA, CS  
 L41 8 S E3-E9  
     E BORINGER/PA, CS  
     E BOEINGER/PA, CS  
     E BOERINGER/PA, CS  
 L42 10 S L37-L39 AND L40, L41  
 L43 1 S L42 AND L35  
 L44 78193 S L9 OR SOLUTE  
 L45 154348 S L8, L44  
 L46 28860 S L45 AND ?SOLVENT?  
 L47 5220 S L45 AND L11-L13  
 L48 28922 S L17, L46, L47  
 L49 2675 S L48 AND (GAS OR GASEOUS)  
 L50 466 S L48 AND GASES+NT/CT  
 L51 988 S L48 AND L4  
 L52 3539 S L24, L49-L51  
 L53 123 S L52 AND (EXPAND? OR EXPANSION?)  
 L54 6 S L52 AND (RETRACT? OR CONTRACT?)  
 L55 114 S L52 AND PRECIPITAT?  
 L56 341 S L52 AND EXTRACT?  
 L57 152 S L52 AND COAT?  
 L58 362 S L52 AND RETENT?  
 L59 64 S L52 AND (?FILTR? OR ?FILTER?)  
 L60 224 S L52 AND ?CRYST?  
 L61 3 S L54 AND L53, L55-L60  
 L62 2 S L61 NOT LITHIUM/TI  
 L63 2 S L37-L39 AND L52  
 L64 1 S L42 AND L52  
 L65 3 S L43, L62-L64  
 L66 9 S L42 NOT L65  
     SEL DN AN 1-6  
 L67 6 S L66 AND E1-E18  
 L68 9 S L65, L67 AND L2-L67  
 L69 594 S L48 AND (SUPERCITIC? OR SUPER CRITIC?) () FLUID?  
 L70 45 S L69 AND (EXPAND? OR EXPANSION?)  
 L71 0 S L70 AND (RETRACT? OR CONTRACT?)  
 L72 26 S L70 AND (EXTRACT? OR COAT? OR RETENT? OR ?CRYST?)  
 L73 915 S L48 AND (SUPERCITIC? OR SUPER CRITIC?)  
 L74 3808 S L73, L52  
 L75 142 S L74 AND (EXPAND? OR EXPANSION?)  
 L76 9 S L75 AND (CONTRACT? OR RETRACT? OR RETENTION?)

SEL DN AN 2  
 L77 1 S E19-E21  
 L78 9 S L68,L77  
 L79 139 S L75 NOT L78  
 L80 63 S L79 AND (?CRYSTAL? OR PRECIPITATE? OR EXTRACT? OR COAT? OR ?FILTR  
 L81 72 S L78,L80  
 L82 30 S L37-L39 NOT L81  
 SEL DN AN 2  
 L83 1 S E22-E24  
 L84 73 S L81,L83  
 L85 64 S L84 AND (?SOLUTE? AND ?SOLVENT?)  
 L86 56 S L85 AND (GAS OR GASEOUS OR L4)  
 L87 45 S L85 AND (SUPERCritical? OR SUPER CRITICAL?)  
 L88 64 S L86,L87  
 L89 9 S L84 NOT L88  
 SEL DN AN 3 9  
 L90 7 S L89 NOT E25-E30  
 L91 71 S L88,L90  
 L92 64 S L91 AND EXPAN?  
 L93 20 S L92 AND (RETRACT? OR CONTRACT? OR RETENTION? OR EXTRACT?)  
 L94 44 S L92 NOT L93  
 L95 27 S L90,L93  
 L96 44 S L91 NOT L95  
 L97 44 S L94,L96

FILE 'REGISTRY' ENTERED AT 14:49:58 ON 08 MAR 2004

FILE 'HCAPLUS' ENTERED AT 14:50:04 ON 08 MAR 2004  
 L98 27 S L95 AND L2-L97

FILE 'REGISTRY' ENTERED AT 14:51:01 ON 08 MAR 2004

=> fil hcaplus  
 FILE 'HCAPLUS' ENTERED AT 14:51:05 ON 08 MAR 2004  
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FILE COVERS 1907 - 8 Mar 2004 VOL 140 ISS 11  
 FILE LAST UPDATED: 5 Mar 2004 (20040305/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d 198 all hitstr tot

L98 ANSWER 1 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:875319 HCAPLUS  
 DN 139:354439  
 ED Entered STN: 07 Nov 2003  
 TI Method for reduction of residual organic solvent in carbomer

IN Forness, Cecile; Horhota, Stephen T.; Saim, Said;  
 Bochniak, David  
 PA Boehringer Ingelheim Pharmaceuticals, Inc., USA  
 SO PCT Int. Appl., 24 pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 IC ICM C08F006-00  
 ICS C08F006-28; B01D011-02; A61K009-00  
 CC 63-5 (Pharmaceuticals)  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2003091290	A1	20031106	WO 2003-US12403	20030421
	W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			US 2003211159	A1 20031113 US 2003-419436 20030421
PRAI	US 2002-374919P	P	20020423		
AB	The method with effective for the reduction of residual organic solvent in carbomer down to the ppm level (e.g., ≤30 ppm), comprises exposing a carbomer (Carbomer 934P) containing residual organic solvent (e.g., benzene) to a gaseous fluid (e.g., CO <sub>2</sub> ) in which the residual organic solvent is soluble and under conditions sufficient to extract at least some of the residual organic solvent from the carbomer. A pharmaceutical suspensions contain the carbomers treated by the method and a therapeutically active agent.				
ST	carbomer residual org solvent redn				
IT	Vasodilators (diuretic, therapeutically active agent; method for reduction of residual organic solvent in carbomer)				
IT	Drugs (method for reduction of residual organic solvent in carbomer)				
IT	Analgesics				
	Anesthetics				
	Anti-inflammatory agents				
	Antibiotics				
	Anticoagulants				
	Antihistamines				
	Antimicrobial agents				
	Antioxidants				
	Antipsychotics				
	Antitumor agents				
	Antiviral agents				
	Decongestants				
	Fungicides				
	Hypnotics and Sedatives				
	Immunosuppressants				
	Nervous system stimulants				
	Thrombolytics (therapeutically active agent; method for reduction of residual organic solvent in carbomer)				
IT	Amino acids, biological studies				
	Hormones, animal, biological studies				

Minerals, biological studies  
 Neurotransmitters  
 Nucleotides, biological studies  
 Peptides, biological studies  
 Proteins  
 Vitamins  
 RL: BUU (Biological use, unclassified); BIOL (Biological study); USES (Uses)  
     (therapeutically active agent; method for reduction of residual organic solvent in carbomer)

IT 57916-92-4, Carbomer 934P  
 RL: BUU (Biological use, unclassified); BIOL (Biological study); USES (Uses)  
     (method for reduction of residual organic solvent in carbomer)

IT 79-10-7DP, Acrylic acid, polymers 9003-97-8P, Polycarbophil 9007-16-3P, Carbomer 934 9062-04-8P, Carbomer 941 76050-42-5P, Carbomer 940 96827-24-6P, Carbomer 1342 126040-58-2P, Calcium polycarbophil  
 RL: BUU (Biological use, unclassified); PUR (Purification or recovery); BIOL (Biological study); PREP (Preparation); USES (Uses)  
     (method for reduction of residual organic solvent in carbomer)

IT 71-23-8, Propanol, uses 71-36-3, Butanol, uses 74-84-0, Ethane, uses 74-85-1, Ethylene, uses 74-98-6, Propane, uses 75-28-5, Isobutane 75-46-7, Trifluoromethane 75-73-0, Tetrafluoromethane 95-47-6, o-Xylene, uses 106-97-8, Butane, uses 110-82-7, Cyclohexane, uses 115-07-1, Propylene, uses 115-11-7, Isobutene, uses 124-38-9, Carbon dioxide, uses 2551-62-4, Sulfur hexafluoride 7664-41-7, Ammonia, uses 7732-18-5, Water, uses 10024-97-2, Nitrous oxide, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
     (method for reduction of residual organic solvent in carbomer)

IT 56-23-5, Carbon tetrachloride, processes 64-17-5, Ethanol, processes 67-56-1, Methanol, processes 67-63-0, Isopropanol, processes 67-64-1, Acetone, processes 67-66-3, Chloroform, processes 67-68-5, Dimethyl sulfoxide, processes 71-43-2, Benzene, processes 75-09-2, Methylene chloride, processes 75-35-4, 1,1-Dichloroethene, processes 79-01-6, Trichloroethylene, processes 107-06-2, 1,2-Dichloroethane, processes 108-88-3, Toluene, processes 108-95-2, Phenol, processes 110-54-3, Hexane, processes 123-91-1, 1,4-Dioxane, processes 141-78-6, Ethyl acetate, processes  
 RL: REM (Removal or disposal); PROC (Process)  
     (residual; method for reduction of residual organic solvent in carbomer)

IT 5534-09-8, Beclomethasone dipropionate 9004-10-8, Insulin, biological studies 13392-18-2, Fenoterol 18559-94-9, Albuterol 22254-24-6, Ipratropium bromide 30286-75-0, Oxytropium bromide 37148-27-9, Clenbuterol 51022-70-9, Albuterol sulfate 71125-38-7, Meloxicam 136310-93-5, Tiotropium bromide  
 RL: BUU (Biological use, unclassified); BIOL (Biological study); USES (Uses)  
     (therapeutically active agent; method for reduction of residual organic solvent in carbomer)

RE.CNT 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 RE  
 (1) Basf Ag; WO 9720866 A 1997 HCPLUS  
 (2) Boehringer Ingelheim Pharma; WO 9909990 A 1999 HCPLUS  
 (3) Bresciani, A; US 5093472 A 1992 HCPLUS

IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
     (method for reduction of residual organic solvent in carbomer)

RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 2 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
AN 2003:666885 HCAPLUS  
DN 140:64873  
ED Entered STN: 27 Aug 2003  
TI Engineering micronization and **coating** applications with dense phase **carbon dioxide**  
AU Subramaniam, Bala  
CS Department of Chemical & Petroleum Engineering, The University of Kansas, Lawrence, KS, 66045-7609, USA  
SO Polymeric Materials Science and Engineering (2003), 89, 678  
CODEN: PMSEDG; ISSN: 0743-0515  
PB American Chemical Society  
DT Journal; (computer optical disk)  
LA English  
CC 63-5 (Pharmaceuticals)  
AB A process for producing and harvesting drug particles on a continuous basis using **supercrit. Carbon dioxide** (**scCO<sub>2</sub>**) as an **antisolvent**, and a Wurster-type **coater** employing **scCO<sub>2</sub>** as the fluidizing medium and **antisolvent** are described. Particle micronization with **scCO<sub>2</sub>** allows for reproducible crystal formation with the potential for increased surface area and dissoln. rates. **Coating** with dense phase **CO<sub>2</sub>** allows the use of traditional organic soluble **coatings** with complete solvent recovery and virtually no atmospheric emissions. For particle micronization, ultrasonic energy is used to form droplets of drug solution. The **scCO<sub>2</sub>** selectively **exts.** the **solvent** from the droplets, **precipitating** the drug. The effluent from the **precipitation** chamber is led to a second high-pressure vessel where the particles are separated from the **solvent-laden scCO<sub>2</sub>**. The micronization of several drugs including proteins and anti-cancer agents will be presented including anal. results such as the particle-size distribution, crystallinity, and residual **solvent** content. Advantages include the continuous production of virtually **solvent-free** drug particles in a narrow size range, **CO<sub>2</sub>** recycling, **solvent** recovery and ease of process scalability. For **coating** applications, glass inner and outer columns are housed in a high-pressure chamber in which dense phase **CO<sub>2</sub>** is used to fluidize the substrates. The **CO<sub>2</sub>** also removes the **solvent** from the **coating** solution sprayed on the substrates, thereby **precipitating** the **coating**. The system was used to **coat** a variety of substrates including tablets and stents for controlled release applications. This process **expands** the range of substrate/**coating** combinations possible with the Wurster **coater**, making it feasible to **coat** water-soluble substrates with solutes sprayed from organic solns.  
ST **supercrit carbon dioxide** fluidizing medium  
**antisolvent coating**; **dense phase carbon dioxide** drug micronization  
IT **Solvents**  
(**antisolvents**; engineering micronization and **coating** applications with dense phase **carbon dioxide**)  
IT **Coating materials**  
(drug; engineering micronization and **coating** applications with dense phase **carbon dioxide**)  
IT **Antitumor agents**  
(engineering micronization and **coating** applications with dense phase **carbon dioxide**)  
IT **Proteins**  
RL: PEP (Physical, engineering or chemical process); PYP (Physical

process); PROC (Process)  
 (engineering micronization and **coating** applications with  
 dense phase **carbon dioxide**)

IT Pulverization  
 (micronization; engineering micronization and **coating**  
 applications with dense phase **carbon dioxide**)

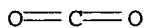
IT Drugs  
 (particles; engineering micronization and **coating**  
 applications with dense phase **carbon dioxide**)

IT 124-38-9, **Carbon dioxide**, biological studies  
 RL: BUU (Biological use, unclassified); BIOL (Biological study); USES  
 (Uses)  
 (**supercrit.**; engineering micronization and **coating**  
 applications with dense phase **carbon dioxide**)

IT 124-38-9, **Carbon dioxide**, biological studies  
 RL: BUU (Biological use, unclassified); BIOL (Biological study); USES  
 (Uses)  
 (**supercrit.**; engineering micronization and **coating**  
 applications with dense phase **carbon dioxide**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L98 ANSWER 3 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:636787 HCAPLUS  
 ED Entered STN: 15 Aug 2003  
 TI Engineering micronization and **coating** applications with dense  
 phase **carbon dioxide**  
 AU Subramaniam, Bala  
 CS Department of Chemical & Petroleum Engineering, The University of Kansas,  
 Lawrence, KS, 66045-7609, USA  
 SO Abstracts of Papers, 226th ACS National Meeting, New York, NY, United  
 States, September 7-11, 2003 (2003), PMSE-405 Publisher: American Chemical  
 Society, Washington, D. C.  
 CODEN: 69EKY9  
 DT Conference; Meeting Abstract  
 LA English  
 AB A process for producing and harvesting drug particles on a continuous  
 basis using **supercrit. carbon dioxide**  
 (scCO<sub>2</sub>) as an **antisolvent**, and a Wurster-type **coater**  
 employing scCO<sub>2</sub> as the fluidizing medium and **antisolvent** are  
 described. Particle micronization with scCO<sub>2</sub> allows for reproducible  
 crystal formation with the potential for increased surface area  
 and dissoln. rates. **Coating** with dense phase CO<sub>2</sub>  
 allows the use of traditional organic soluble **coatings** with complete  
**solvent** recovery and virtually no atmospheric emissions. For particle  
 micronization, ultrasonic energy is used to form droplets of drug solution  
 The scCO<sub>2</sub> selectively **exts.** the **solvent** from the  
 droplets, **precipitating** the drug. The effluent from the **precipitation**  
 chamber is led to a second high-pressure vessel where the particles are  
 separated from the **solvent**-laden scCO<sub>2</sub>. The micronization of  
 several drugs including proteins and anti-cancer agents will be presented  
 including anal. results such as the particle-size distribution,  
**crystallinity**, and residual **solvent** content. Advantages  
 include the continuous production of virtually **solvent-free** drug  
 particles in a narrow size range, CO<sub>2</sub> recycling, **solvent**  
 recovery and ease of process scalability. For **coating**  
 applications, glass inner and outer columns are housed in a high-pressure  
 chamber in which dense phase CO<sub>2</sub> is used to fluidize the

substrates. The CO<sub>2</sub> also removes the solvent from the coating solution sprayed on the substrates, thereby precipitating the coating. The system was used to coat a variety of substrates including tablets and stents for controlled release applications. This process expands the range of substrate/coating combinations possible with the Wurster coater, making it feasible to coat water-soluble substrates with solutes sprayed from organic solns.

L98 ANSWER 4 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:424564 HCPLUS  
 DN 138:390844  
 ED Entered STN: 04 Jun 2003  
 TI Preparation of superfine particles using fast expanding supercritical solution  
 IN Yin, Enhua  
 PA Huayu New-Type Electronics Material Co., Ltd., Peop. Rep. China  
 SO Faming Zhuanli Shenqing Gongkai Shuomingshu, 7 pp.  
 CODEN: CNXXEV

DT Patent  
 LA Chinese  
 IC ICM B01D009-02  
 ICS B01D011-00  
 CC 63-1 (Pharmaceuticals)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	CN 1344578	A	20020417	CN 2000-124564	20000922
PRAI	CN 2000-124564		20000922		

AB The method comprises dissolving and swelling solute (such as aspirin, polylactic acid, stimulants, antiphlogistic, contraceptive, release-controlling agent, other polymer, or pigment) in CO<sub>2</sub> (or metallic oxide in water) under supercrit. condition, ejecting, filtering, and settling. The equipment consists of solvent tank, high-pressure pump, heat exchanger, extraction reactor, pressure reactor, elec. control unit, nozzle, filter, flow gauge, and refrigerating machine.

ST fast expanding superfine particle supercrit soln

IT Supercritical fluids

(fast expanding; preparation of superfine particles using fast expanding supercrit. solution)

IT Drug delivery systems

(particles, superfine; preparation of superfine particles using fast expanding supercrit. solution)

IT Anti-inflammatory agents

Contraceptives

Nervous system stimulants

(preparation of superfine particles using fast expanding supercrit. solution)

IT 124-38-9, Carbon dioxide, processes

RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)

(preparation of superfine particles using fast expanding supercrit. solution)

IT 50-78-2, Aspirin 26023-30-3, Poly[oxy(1-methyl-2-oxo-1,2-ethanediyl)] 26100-51-6, Polylactic acid

RL: PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)

(preparation of superfine particles using fast expanding supercrit. solution)

IT 124-38-9, Carbon dioxide, processes

RL: PEP (Physical, engineering or chemical process); PYP (Physical

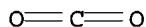
process); PROC (Process)  
 (preparation of superfine particles using fast **expanding supercrit.** solution)

RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O—C—O

L98 ANSWER 5 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:421466 HCPLUS  
 DN 140:133534  
 ED Entered STN: 03 Jun 2003  
 TI Application of dense **gas** techniques for the production of fine particles  
 AU Foster, Neil R.; Dehghani, Fariba; Charoenchaitrakool, Kiang M.; Warwick, Barry  
 CS School of Chemical and Industrial Chemistry, University of New South Wales, NSW 2052, Australia  
 SO PharmSci (2003), 5(2), No pp. given  
 CODEN: PHARFY; ISSN: 1522-1059  
 URL: <http://www.pharmsci.org/view.asp?path=ps0502/ps050211/ps050211.xml&pd=f=yes>  
 PB American Association of Pharmaceutical Scientists  
 DT Journal; (online computer file)  
 LA English  
 CC 63-5 (Pharmaceuticals)  
 AB The feasibility of using dense **gas** techniques such as rapid **expansion of supercrit.** solns. (RESS) and aerosol **solvent extraction** system (ASES) for micronization of pharmaceutical compds. is demonstrated. The chiral nonsteroidal anti-inflammatory racemic ibuprofen is soluble in **carbon dioxide** at 35°C and pressures above 90 bar. The particle size decreased to less than 2 µm while the degree of **crystallinity** was slightly decreased when processed by RESS. The dissoln. rate of the ibuprofen (a poorly water-soluble compound) was significantly enhanced after processing by RESS. The nonsteroidal anti-inflammatory drug Cu<sub>2</sub>(indomethacin)4L<sub>2</sub>(Cu-Indo); (L = DMF [DMF]), which possessed very low solubility in **supercrit.** CO<sub>2</sub>, was successfully micronized by ASES at 25°C and 68.9 bar using DMF as the **solvent** and CO<sub>2</sub> as the **antisolvent**. The concentration of **solute** dramatically influenced the **precipitate** characteristics. The particles obtained from the ASES process were changed from bipyramidal to spherical, with particle size less than 5 µm, as the concentration increased from 5 to 100 mg/g. A further increase in **solute** concentration to 200 mg/g resulted in large porous spheres, between 20 and 50 µ, when processing Cu-Indo by the ASES method. The dissoln. rate of the micronized Cu-Indo was significantly higher than the com. product.  
 ST ibuprofen micronization aerosol **solvent extn**  
**supercrit** fluid dissoln **recrystn**; copper  
 indomethacin micronization aerosol **solvent extn**  
**supercrit** fluid dissoln; ophthalmic suspension copper  
 indomethacin dissoln dense **gas** particle size  
 IT **Solvent extraction**  
 (aerosol; application of dense **gas** techniques for the production of fine particles)  
 IT **Crystallinity**  
 Dissolution  
 Particle shape  
 Particle size

Particle size distribution  
 Recrystallization  
 Solubility  
 Supercritical fluids  
 (application of dense **gas** techniques for the production of fine particles)  
 IT Pulverization  
 (micronization; application of dense **gas** techniques for the production of fine particles)  
 IT Drug delivery systems  
 (suspensions, ophthalmic; application of dense **gas** techniques for the production of fine particles)  
 IT 124-38-9, Carbon dioxide, uses 151-21-3,  
 Sodium lauryl sulfate, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (application of dense **gas** techniques for the production of fine particles)  
 IT 15687-27-1, Ibuprofen 221357-17-1  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)  
 (application of dense **gas** techniques for the production of fine particles)  
 RE.CNT 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 RE  
 (1) Bustami, R; Kona 2001, V19, P57 HCPLUS  
 (2) Chroenchaintrakool, M; Ind Eng Chem Res 2000, V39, P4794  
 (3) Falk, R; J Pharm Res 1998, V15, P1233 HCPLUS  
 (4) Lengsfeld, C; J Phys Chem B 2000, V104, P2725 HCPLUS  
 (5) Philips, E; Int J Pharm 1993, V94, P1  
 (6) Reverchon, E; J Supercritic Fluids 2000, V17, P239 HCPLUS  
 (7) Tom, J; Biotechnol Prog 1991, V7, P403 HCPLUS  
 (8) Tom, J; J Aerosol Sci 1991, V22, P555 HCPLUS  
 (9) Warwick, B; Ind Eng Chem Res 2002, V41(8), P1993 HCPLUS  
 (10) Williams, R; WO 02/060411 A2 2002 HCPLUS  
 IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (application of dense **gas** techniques for the production of fine particles)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L98 ANSWER 6 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:287335 HCPLUS  
 DN 138:293388  
 ED Entered STN: 15 Apr 2003  
 TI Spectroscopically Probing Microscopic **Solvent** Properties of Room-Temperature Ionic Liquids with the Addition of **Carbon Dioxide**  
 AU Lu, Jie; Liotta, Charles L.; Eckert, Charles A.  
 CS Schools of Chemical Engineering and Chemistry and Biochemistry and Specialty Separations Center, Georgia Institute of Technology, Atlanta, GA, 30332-0100, USA  
 SO Journal of Physical Chemistry A (2003), 107(19), 3995-4000  
 CODEN: JPCAFH; ISSN: 1089-5639  
 PB American Chemical Society  
 DT Journal  
 LA English

- CC 68-1 (Phase Equilibria, Chemical Equilibria, and Solutions)  
 Section cross-reference(s): 73, 76
- AB Room-temperature ionic liqs. (RTILs) provide an alternative for elimination of solvent emissions to the atmospheric for many reactions, but the subsequent separation of the products by conventional methods can be a challenge. However, the use of supercrit. carbon dioxide (scCO<sub>2</sub>) as an extractant offers potential for a novel class of environmentally benign media for chemical reaction and downstream separation. The authors studied the solvent properties of mixts. of 1-butyl-3-Me imidazolium hexafluorophosphate ([bmim][PF<sub>6</sub>]) and CO<sub>2</sub> as functions of temperature (35-50 °C) and CO<sub>2</sub> pressure (0-230 bar). They report the Kamlet-Taft dipolarity/polarizability parameter, volume expansion, and microviscosity. The results are consistent with a picture of local enhancement of RTIL composition around a chromophore, maintaining solvent strength even at fairly high loadings of CO<sub>2</sub>, whereas the microviscosity in the vicinity of the solute is dramatically reduced, leading to enhanced mass transport and facilitated separation
- ST alkyl imidazolium fluorophosphate carbon dioxide mixt  
 solvent property solvatochromism
- IT Dielectric constant  
 Fluorescence  
 Green chemistry  
 Ionic liquids  
 Mass transfer  
 Microviscosity  
 Polarizability  
 Separation  
**Solvatochromism**  
 (solvent properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with carbon dioxide as studied by solvatochromic and fluorescence probes)
- IT Expansion  
 (volume; solvent properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with carbon dioxide as studied by solvatochromic and fluorescence probes)
- IT 100-23-2, N,N-Dimethyl-4-nitroaniline 58293-56-4, DCVJ  
 RL: ARU (Analytical role, unclassified); ANST (Analytical study)  
 (solvent properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with carbon dioxide as studied by solvatochromic and fluorescence probes)
- IT 124-38-9, Carbon dioxide, properties  
 174501-64-5, 1-Butyl-3-methyl imidazolium hexafluorophosphate  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)  
 (solvent properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with carbon dioxide as studied by solvatochromic and fluorescence probes)
- RE.CNT 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD
- RE
- (1) Aki, S; Chem Commun 2001, P413 HCAPLUS
  - (2) Baker, S; Green Chem 2002, V4, P165 HCAPLUS
  - (3) Baker, S; J Phys Chem B 2001, V105, P9663 HCAPLUS
  - (4) Blanchard, L; Ind Eng Chem Res 2001, V40, P287 HCAPLUS
  - (5) Blanchard, L; J Phys Chem B 2001, V105, P2437 HCAPLUS
  - (6) Blanchard, L; Nature 1999, V399, P28
  - (7) Bosmann, A; Angew Chem 2001, V113, P2769
  - (8) Brennecke, J; AIChE J 2001, V47, P2384 HCAPLUS
  - (9) Brown, R; J Am Chem Soc 2001, V123, P1254 HCAPLUS
  - (10) Carmichael, A; J Phys Org Chem 2000, V13, P591
  - (11) Cull, S; Biotechnol Bioeng 2000, V69, P227 HCAPLUS
  - (12) Eckert, C; Nature 1996, V383, P313 HCAPLUS

- (13) Ely, J; J Chem Thermodyn 1989, V21, P879 HCAPLUS  
 (14) Gordon, C; Appl Catal A 2001, V222, P101 HCAPLUS  
 (15) Hou, Z; New J Chem 2002, V26, P1246 HCAPLUS  
 (16) Huddleston, J; Green Chem 2001, V3, P156 HCAPLUS  
 (17) Hyk, W; J Phys Chem B 1998, V102, P577 HCAPLUS  
 (18) Kamlet, M; J Am Chem Soc 1977, V99, P6027 HCAPLUS  
 (19) Kazarian, S; Chem Commun 2000, P2047 HCAPLUS  
 (20) Kung, C; Biochemistry 1986, V25, P6114 HCAPLUS  
 (21) Kung, C; Biochemistry 1989, V28, P6678 HCAPLUS  
 (22) Liu, F; Chem Commun 2001, P433 HCAPLUS  
 (23) Loutfy, R; J Phys Chem 1982, V86, P4205 HCAPLUS  
 (24) Loutfy, R; Pure Appl Chem 1986, V58, P1239 HCAPLUS  
 (25) Lozano, P; Chem Commun 2002, P692 HCAPLUS  
 (26) Marcus, Y; Chem Soc Rev 1993, P409 HCAPLUS  
 (27) Muldoon, M; J Chem Soc, Perkin Trans 2001, V2, P433  
 (28) Nara, S; Tetrahedron Lett 2002, V43, P2979 HCAPLUS  
 (29) Ngo, H; Thermochim Acta 2000, V357, P97  
 (30) Olivier-Bourbigou, H; J Mol Catal 2002, V3484, P1  
 (31) Peng, J; New J Chem 2001, V25, P639 HCAPLUS  
 (32) Scurto, A; J Am Chem Soc 2002, V124, P10276 HCAPLUS  
 (33) Sellin, M; Chem Commun 2001, P781 HCAPLUS  
 (34) Wasserscheid, P; Ionic Liquids in Synthesis 2002  
 (35) Welton, T; Chem Rev 1999, V99, P2071 HCAPLUS

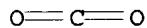
IT 124-38-9, Carbon dioxide, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)

(solvent properties of 1-butyl-3-Me imidazolium hexafluorophosphate mixts. with carbon dioxide as studied by solvatochromic and fluorescence probes)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L98. ANSWER 7 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:281854 HCAPLUS

DN 138:292772

ED Entered STN: 11 Apr 2003

TI Powder processing with pressurized gaseous fluids

IN Saim, Said; Horhota, Stephen; Koenig, Kenneth James; Bochniak, David Joseph

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO U.S. Pat. Appl. Publ., 37 pp.

CODEN: USXXCO

DT Patent

LA English

IC ICM B01D011-00

NCL 210634000; 210638000; 210644000; 210669000; 210702000; 210806000

CC 63-6 (Pharmaceuticals)

Section cross-reference(s): 48

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2003066800	A1	20030410	US 2002-268879	20021010
	WO 2003030871	A1	20030417	WO 2002-US32303	20021010
	W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,			

UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRAI US 2001-328301P P 20011010

AB Disclosed is a method of small particle precipitation, retention and dispersion of

a solid or semi-solid material onto or into a carrier material. In the method, **solute** particles are precipitated from a pressurized gaseous fluid solution or a liquid solution and effectively retained and dispersed within

a carrier material. The technique can be advantageously used in pharmaceutical processing to produce a blend of solid or semi-solid material particles and carrier material, a granulation of the solid or semi-solid material particles with carrier material partially or fully coated with the solid and/or semi-solid material particles.

ST pharmaceutical particle pptn retention dispersion solid semisolid; pressurized gaseous fluid particle formation

IT Drug delivery systems

(capsules; method of particle precipitation and retention in carrier for processing)

IT Supercritical fluids

(for precipitation of **solute** in processing pharmaceutical particles)

IT Precipitation (chemical)

(powder processing with pressurized gaseous fluids)

IT Drug delivery systems

(tablets; method of particle precipitation and retention in carrier for processing)

IT 22254-24-6, Ipratropium bromide 30286-75-0, Oxytropium bromide 136310-93-5, Tiotropium bromide 174484-41-4, Tipranavir

RL: DMA (Drug mechanism of action); BIOL (Biological study)  
(active material in processing pharmaceutical particles)

IT 9003-70-7, Polystyrene divinyl benzene 64044-51-5, Lactose monohydrate

RL: DMA (Drug mechanism of action); BIOL (Biological study)  
(carrier for processing pharmaceutical particles)

IT 74-84-0, Ethane, uses 74-85-1, Ethylene, uses 74-98-6, Propane, uses 75-28-5, Isobutane 75-46-7, Trifluoromethane 106-97-8, Butane, uses 109-66-0, Pentane, uses 115-07-1, Propylene, uses 124-38-9, Carbon dioxide, uses 2551-62-4, Sulfur hexafluoride 10024-97-2, Nitrous oxide, uses

RL: TEM (Technical or engineered material use); USES (Uses)

(for **solute** precipitation in producing of pharmaceutical particles)

IT 124-38-9, Carbon dioxide, uses

RL: TEM (Technical or engineered material use); USES (Uses)

(for **solute** precipitation in producing of pharmaceutical particles)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 8 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:227421 HCAPLUS

DN 138:227493

ED Entered STN: 25 Mar 2003

TI Solubilities of Imipramine HCl in **Supercritical Carbon Dioxide**

AU Jara-Morante, Eliana; Suleiman, David; Estevez, L. Antonio

CS Department of Chemical Engineering, University of Puerto Rico, Mayagueez,

00681-9046, P. R.

SO Industrial & Engineering Chemistry Research (2003), 42(8), 1821-1823  
CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

LA English

CC 68-1 (Phase Equilibria, Chemical Equilibria, and Solutions)  
Section cross-reference(s): 45, 47, 63

AB The solubility of imipramine hydrochloride (I) in **supercrit. carbon dioxide** has been measured exptl. by a gravimetric technique. An ISCO **extraction** apparatus was modified to carry out the measurements. It consists of a syringe pump, a thermostatic chamber, an equilibrium cell, a variable-flow-rate restrictor, and an ice trap. Expts. were conducted by allowing the **supercrit. carbon dioxide** to slowly flow through the cell, where I had been previously loaded. The pressure was kept constant, controlled by the pump, and so was the flow rate, controlled by the restrictor. The amount of **solute** collected in the trap was measured in two different ways for consistency: gravimetrically and by dissolving the **solute** collected in methanol and measuring the concentration spectrophotometrically. The amount of **solvent** was measured by the difference in volume readings in the syringe pump (calculating the d. of **carbon dioxide** at the pump conditions); this value was also determined by measuring an average flow rate of the **expanded solvent** and the time of the run. A total of 52 measurements were done. Two five-point isotherms, at 40 and 50 °C, were obtained for pressures ranging from 30 to 50 MPa. Measured solubilities were within the range (5-10) + 10-6 mole fraction. These are the only published data for this system.

ST imipramine hydrochloride soly **supercrit carbon dioxide**

IT Extraction apparatus  
(**extraction** set for determination of imipramine hydrochloride solubility in **supercrit. carbon dioxide**)

IT Solubility  
(imipramine hydrochloride solubility in **supercrit. carbon dioxide**)

IT Solvents  
(**supercrit.**; imipramine hydrochloride solubility in **supercrit. carbon dioxide**)

IT 113-52-0, Imipramine hydrochloride 124-38-9, Carbon dioxide, properties  
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)  
(imipramine hydrochloride solubility in **supercrit. carbon dioxide**)

RE.CNT 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Jara-Morante, E; M S Thesis, University of Puerto Rico 1999
- (2) Larson, K; Biotechnol Prog 1986, V2(2), P73 HCAPLUS
- (3) Macnaughton, S; J Chem Eng Data 1996, V41, P1083 HCAPLUS
- (4) Maxwell, R; Proc 2nd Int Symp Supercrit Fluids 1991, P143
- (5) Schaeffer, S; Supercritical Fluid Science and Technology; ACS Symposium Series 406 1989, P416 HCAPLUS
- (6) Ting, S; Ind Eng Chem Res 1993, V32, P1471 HCAPLUS
- (7) Vandana, V; Fluid Phase Equilib 1997, V135, P83 HCAPLUS
- (8) Yun, S; Ind Eng Chem Res 1991, V30(11), P2476 HCAPLUS

IT 124-38-9, Carbon dioxide, properties  
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)  
(imipramine hydrochloride solubility in **supercrit. carbon dioxide**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 9 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
AN 2002:951622 HCAPLUS  
DN 139:154684  
ED Entered STN: 17 Dec 2002  
TI Crystal doping aided by rapid expansion of supercritical solutions  
AU Vemavarapu, Chandra; Mollan, Matthew J.; Needham, Thomas E.  
CS Pharmaceutical Sci., pfizer Global R&D, Ann Arbor, MI, 48105, USA  
SO AAPS PharmSciTech (2002), 3(4), No pp. given  
CODEN: AAPHFZ; ISSN: 1522-1059  
URL: [http://www.aapspharmscitech.org/scientificjournals/pharmscitech/volume\\_e3issue4/pt030429/pt030429.pdf](http://www.aapspharmscitech.org/scientificjournals/pharmscitech/volume_e3issue4/pt030429/pt030429.pdf)

PB American Association of Pharmaceutical Scientists  
DT Journal; (online computer file)  
LA English  
CC 63-5 (Pharmaceuticals)  
AB The purpose of this study was to test the utility of rapid expansion of supercrit. solution (RESS) based cocrystns. in inducing polymorph conversion and crystal disruption of chlorpropamide (CPD). CPD crystals were recrystd. by the RESS process utilizing supercrit. carbon dioxide as the solvent. The supercrit. region investigated for solute extn . ranged from 45 to 100°C and 2000 to 8000 psi. While pure solute recrystn. formed stage I of these studies, stage II involved recrystn. of CPD in the presence of urea (model impurity). The composition, morphol., and crystallinity of the particles thus produced were characterized utilizing techniques such as microscopy, thermal anal., x-ray powder diffractometry, and HPLC. Also, comparative evaluation between RESS and evaporative crystallization from liquid solvents was performed. RESS recrystns. of com. available CPD (form A) resulted in polymorph conversion to metastable forms C and V, depending on the temperature and pressure of the recrystg . solvent. Cocrystn. studies revealed the formation of eutectic mixts. and solid solns. of CPD + urea. Formation of the solid solns. resulted in the crystal disruption of CPD and subsequent amorphous conversion at urea levels higher than 40% wt/weight Consistent with these results were the redns. in m.p. (up to 9°C) and in the ΔHfvalues of CPD (up to 50%). SEM revealed a particle size reduction of up to an order of magnitude upon RESS processing. Unlike RESS, recrystns. from liquid organic solvents lacked the ability to affect polymorphic conversions. Also, the incorporation of urea into the lattice of CPD was found to be inadequate. In providing the ability to control both the particle and crystal morphologies of active pharmaceutical ingredients, RESS proved potentially advantageous to crystal engineering. Rapid crystallization kinetics were found vital in making RESS-based doping superior to conventional solvent -based cocrystns.  
ST chlorpropamide crystal doping supercrit soln  
IT Crystallization  
    (cocrystn.; crystal doping aided by rapid expansion of supercrit. solns.)  
IT Polymorphism (crystal)  
    Supercritical fluids  
    (crystal doping aided by rapid expansion of supercrit. solns.)

- IT 57-13-6, Urea, uses  
 RL: MOA (Modifier or additive use); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses)  
 (crystal doping aided by rapid expansion of supercrit. solns.)
- IT 94-20-2, Chlorpropamide  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)  
 (crystal doping aided by rapid expansion of supercrit. solns.)
- IT 124-38-9, Carbon dioxide, processes  
 RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)  
 (crystal doping aided by rapid expansion of supercrit. solns.)

RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Aal-Saieq, S; Pharm Acta Helv 1982, V57(1), P8
- (2) Addadi, L; J Am Chem Soc 1982, V104, P4610 HCPLUS
- (3) Behme, R; J Pharm Sci 1991, V80(10), P986 HCPLUS
- (4) Burger, A; Sci Pharm 1975, V43, P152 HCPLUS
- (5) Burt, H; Int J Pharm 1981, V9, P137 HCPLUS
- (6) Chow, A; Int J Pharm 1995, V126, P21 HCPLUS
- (7) Cram, D; Science 1988, V240, P760 HCPLUS
- (8) Dandge, D; Indus Eng Chem Prod Res Dev 1985, V24, P162 HCPLUS
- (9) Davey, R; J Am Chem Soc 1997, V119(7), P1767 HCPLUS
- (10) De Villiers, M; Acta Pharm 1999, V49, P79 HCPLUS
- (11) Debenedetti, P; Fluid Phase Equil 1993, V82, P311 HCPLUS
- (12) Dobbs, J; Indus Eng Chem Res 1987, V26, P56 HCPLUS
- (13) Ford, J; J Pharm Pharmacol 1977, V29, P209 HCPLUS
- (14) Gu, C; AAPS PharmSci 2001, V3(3)
- (15) Hyatt, J; J Org Chem 1984, V49, P5097 HCPLUS
- (16) Jane Li, Z; Int J Pharm 1996, V137, P21
- (17) Klarner, F; Tetrahedron 2001, V57, P3673 HCPLUS
- (18) Koo, C; Arch Pharm Res 1980, V3(1), P37 HCPLUS
- (19) Lemieux, R; US 5989451 1999 HCPLUS
- (20) Mohamed, R; ACS Symposium Series 1989, V406, P355 HCPLUS
- (21) Otsuka, M; J Pharm Sci 1995, V84(5), P614 HCPLUS
- (22) Prasad, K; Int J Pharm 2001, V215, P29 HCPLUS
- (23) Simmons, D; Can J Pharm Sci 1973, V8(4), P125 HCPLUS
- (24) Turk, M; J Supercrit Fluids 1999, V15, P79 HCPLUS
- (25) Weissbuch, I; Crystallization Technology Handbook. 2nd ed 2001, P563
- (26) Weissbuch, I; Science 1991, V253, P637 HCPLUS
- (27) Williams-Seton, L; J Pharm Sci 2000, V89(3), P346 HCPLUS
- (28) Yoshihashi, Y; Int J Pharm 2000, V204, P1 HCPLUS
- (29) Yu, L; J Pharm Sci 1995, V84(8), P966 HCPLUS

IT 124-38-9, Carbon dioxide, processes

- RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)  
 (crystal doping aided by rapid expansion of supercrit. solns.)

RN 124-38-9 HCPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

ED Entered STN: 31 Jul 2002  
 TI Process for overcoming drug retention in hard gelatin inhalation capsules  
 AU Saim, Said; Horhota, Stephen T.  
 CS Boehringer Ingelheim Pharmaceuticals, Inc., Ridgefield, CT, 06877,  
 USA  
 SO Drug Development and Industrial Pharmacy (2002), 28(6), 641-654  
 CODEN: DDIPD8; ISSN: 0363-9045  
 PB Marcel Dekker, Inc.  
 DT Journal  
 LA English  
 CC 63-6 (Pharmaceuticals)  
 AB The quantity and consistency of drug delivery from dry powder inhalation devices that incorporate a pre-measured dose in a hard shell capsule of gelatin or other compatible material can be neg. affected by mold release lubricants used in capsule manufacturing. This paper describes a novel process employing supercrit. CO<sub>2</sub> for selective extraction of the fraction of lubricant responsible for the observed high and inconsistent drug retention in capsules and the ensuing lack of reproducibility of drug delivery. The process allows for lubricant removal from seemingly inaccessible interior surfaces of assembled capsule shells without altering the structural or chemical properties of the capsules. Diffusion limitations are overcome through repeated pressure increase and decrease to generate significant convective flow of dissolved lubricant out of the capsule. Drug retention is alleviated only if nearly all the retentive fraction of the lubricant is removed. The effect of extraction with supercrit. CO<sub>2</sub> on the structure of the internal surfaces of the capsules is investigated using SEM. Key performance parameters such as drug and carrier retention and fine particle mass are investigated using simulated inhalation tests.  
 Laboratory and pilot scale extns. yielded similar results.  
 ST drug retention inhaler gelatin capsule lubricant  
 IT Drug delivery systems  
     (capsules; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)  
 IT Medical goods  
     (inhalers; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)  
 IT Lubricants  
     (overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)  
 IT Drug delivery systems  
     (powders, inhalants; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)  
 IT Extraction  
     (supercrit.; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)  
 IT 63-42-3, Lactose 22254-24-6, Ipratropium bromide  
 RL: PRP (Properties); THU (Therapeutic use); BIOL (Biological study); USES (Uses)  
     (overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)  
 IT 124-38-9, Carbon dioxide, biological studies  
 RL: BSU (Biological study, unclassified); BIOL (Biological study)  
     (supercrit.; overcoming drug retention in hard gelatin inhalation capsules by supercrit. fluid extraction)  
 RE.CNT 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 RE  
 (1) Broadhead, J; Pharm Acta Helv 1995, V70, P125 HCPLUS  
 (2) Brown, S; Unpublished Results 1994  
 (3) Clark, A; US 5641510 1997 HCPLUS  
 (4) Ganderton, D; Adv Pharm Sci 1991, V6, P165  
 (5) Horhota, S; US 6228394 HCPLUS  
 (6) Horhota, S; US 6294194 HCPLUS  
 (7) McHugh, M; Supercritical Fluid Extraction, Principles and Practice, 2nd Ed

1993

- (8) Saim, S; Manuscript in Preparation 2001  
 (9) Witek, T; Poster Presentation, American Thoracic Society Meeting 1998  
 (10) Zimon, A; Adhesion of Dust and Powder, 2nd Ed P264  
 IT 124-38-9, Carbon dioxide, biological studies  
     RL: BSU (Biological study, unclassified); BIOL (Biological study)  
       (supercrit.; overcoming drug retention in hard gelatin inhalation  
       capsules by supercrit. fluid extraction)  
 RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

- L98 ANSWER 11 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 2002:267861 HCAPLUS  
 DN 137:125601  
 ED Entered STN: 10 Apr 2002  
 TI Effects of solvent density on retention in gas  
     -liquid chromatography I. Alkanes solutes in polyethylene glycol  
     stationary phases  
 AU Gonzalez, F. R.; Perez-Parajon, J.; Garcia-Dominguez, J. A.  
 CS Instituto de Quimica-Fisica Rocasolano, CSIC, Madrid, 28006, Spain  
 SO Journal of Chromatography, A (2002), 953(1-2), 151-163  
 CODEN: JCRAEY; ISSN: 0021-9673  
 PB Elsevier Science B.V.  
 DT Journal  
 LA English  
 CC 36-5 (Physical Properties of Synthetic High Polymers)  
     Section cross-reference(s): 66, 80  
 AB Gas-liquid chromatog. columns were prepared by coating  
     silica capillaries with poly(oxyethylene) polymers of different mol. mass  
     distributions, in the range of low number-average molar masses, where the d.  
     still varies significantly. A novel, high-temperature, rapid evaporation  
     method was  
     developed and applied to the static coating of the low-mol.-mass  
     stationary phases. The anal. of alkanes retention data from  
     these columns reveals that the dependence of the partition coefficient with the  
     solvent macroscopic d. is mainly due to a variation of entropy.  
     Enthalpies of solute transfer contribute poorly to the observed  
     variations of retention. Since the alkanes solubility diminishes  
     with the increasing solvent d., and this variation is weakly  
     dependent with temperature, it is concluded that the decrease of free-volume in  
     the liquid is responsible for this behavior.  
 ST polyethylene glycol gas liq chromatog density; alkane  
     retention thermodyn property chromatog polyethylene glycol  
 IT Polyoxalkylenes, processes  
     RL: PEP (Physical, engineering or chemical process); POF (Polymer in  
       formulation); PYP (Physical process); TEM (Technical or engineered  
       material use); PROC (Process); USES (Uses)  
       (Carbowax 1000, Carbowax 1540; alkanes solutes in  
       polyethylene glycol stationary phases gas-liquid chromatog.)  
 IT Gas chromatography  
     (alkanes solutes in polyethylene glycol stationary phases  
       gas-liquid chromatog.)  
 IT Alkanes, properties  
     RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP  
       (Physical process); PROC (Process)  
       (alkanes solutes in polyethylene glycol stationary phases  
       gas-liquid chromatog.)  
 IT Thermal expansion

(coefficient; of polyethylene glycol stationary phases on alkanes retention in gas-liquid chromatog.)

IT Heat capacity  
 Partition  
 Transfer enthalpy  
 Transfer entropy  
 (of alkanes solutes in polyethylene glycol stationary phases gas-liquid chromatog.)

IT Density  
 Molecular weight  
 (of polyethylene glycol stationary phases on alkanes retention in gas-liquid chromatog.)

IT 25322-68-3, Carbowax 600  
 RL: PEP (Physical, engineering or chemical process); POF (Polymer in formulation); PYP (Physical process); TEM (Technical or engineered material use); PROC (Process); USES (Uses)  
 (Carbowax 1000, Carbowax 1540; alkanes solutes in polyethylene glycol stationary phases gas-liquid chromatog.)

IT 208196-86-5, HP-Innowax 318235-13-1, AT-wax  
 RL: PEP (Physical, engineering or chemical process); PYP (Physical process); TEM (Technical or engineered material use); PROC (Process); USES (Uses)  
 (alkanes solutes in crosslinked polyethylene glycol stationary phases gas-liquid chromatog.)

IT 7631-86-9, Silica, miscellaneous  
 RL: MSC (Miscellaneous)  
 (alkanes solutes in polyethylene glycol stationary phases gas-liquid chromatog.)

IT 111-84-2, n-Nonane 112-40-3, n-Dodecane 124-18-5, n-Decane 629-50-5,  
 n-Tridecane 629-59-4, n-Tetradecane 1120-21-4, n-Undecane  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)  
 (alkanes solutes in polyethylene glycol stationary phases gas-liquid chromatog.)

RE.CNT 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Anon; HyperChem, Computational Chemistry 1996
- (2) Bailey, F; Polyethylene Oxide 1976
- (3) Ben Naim, A; Solvation Thermodynamics 1987
- (4) Berezkin, V; Adv Chromatogr 2000, V40, P599 HCPLUS
- (5) Berezkin, V; Chromatographia 1985, V20, P482 HCPLUS
- (6) Billmeyer, F; Textbook of Polymer Science 3rd ed 1984
- (7) Elias, H; Macromolecules 2nd ed 1984, V2
- (8) Evans, M; J Chromatogr 1968, V36, P489 HCPLUS
- (9) Fritz, D; Anal Chem 1973, V45, P1175 HCPLUS
- (10) Gonzalez, F; J Chromatogr A 1999, V832, P165 HCPLUS
- (11) Gonzalez, F; J Chromatogr A 1999, V852, P583 HCPLUS
- (12) Gonzalez, F; J Chromatogr A 2000, V873, P209 HCPLUS
- (13) Gonzalez, F; J Chromatogr A 2001, V927, P111 HCPLUS
- (14) Gonzalez, F; J Chromatogr A 2002, V942, P211 HCPLUS
- (15) Grob, K; Making and Manipulating Capillary Columns for Gas Chromatography 1986
- (16) Hesse, P; J Chem Eng Data 1996, V41, P195 HCPLUS
- (17) Hill, F; Ind Eng Chem 1958, V50, P5 HCPLUS
- (18) Hummel, D; IR Spectra of Polymers in the Medium and Long Wavelength Regions 1966
- (19) Kersten, B; J Chromatogr 1987, V399, P1 HCPLUS
- (20) Kersten, B; J Chromatogr 1989, V468, P235
- (21) Klein, J; Makromol Chem 1980, V181, P1237 HCPLUS
- (22) Kramer, J; J Chromatogr Sci 1985, V23, P54 HCPLUS
- (23) Martin, M; J Phys Chem B 1999, V103, P2977 HCPLUS
- (24) Martire, D; Anal Chem 1974, V46, P626 HCPLUS
- (25) Miller, R; J Polym Sci 1959, V34, P161 HCPLUS

- (26) Peebles, W; Molecular Weight Distributions in Polymers 1968  
 (27) Poole, C; Chromatographia 1992, V34, P281 HCAPLUS  
 (28) Poole, C; J Chromatogr A 2000, V898, P211 HCAPLUS  
 (29) Prigogine, I; Molecular Theory of Solutions 1957  
 (30) Sandler, S; Polymer Synthesis 1974, V1 and 3  
 (31) Sandra, P; J Chromatogr 1989, V411, P63  
 (32) Schweitzer, O; Chemische Berichte 1929, V62, P2395  
 (33) Staudinger, H; From Organic Chemistry to Macromolecules 1970  
 (34) Taleb-Bendiab, S; J Chromatogr 1975, V107, P15 HCAPLUS  
 (35) Thomas, D; J Polym Sci 1960, V42, P195 HCAPLUS  
 (36) Vera, J; Chem Eng J 1972, V3, P1 HCAPLUS  
 (37) Xu, B; J Chromatogr 1988, V445, P1 HCAPLUS

L98 ANSWER 12 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:703010 HCAPLUS

DN 135:247285

ED Entered STN: 26 Sep 2001

TI Method for extraction and reaction using supercritical fluids

IN Horhota, Stephen T.; Saim, Said

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO U.S., 17 pp., Cont.-in-part of U.S. 6,228,394.

CODEN: USXXAM

DT Patent

LA English

IC ICM A61K009-64

ICS A61K009-48; F26B003-00

NCL 424456000

CC 63-8 (Pharmaceuticals)

FAN.CNT 3

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 6294194	B1	20010925	US 2000-517883	20000303
→	US 6228394	B1	20010508	US 1998-157267	19980921 ←
ZA	9809261	A	19990531	ZA 1998-9261	19981012
WO	2001066214	A1	20010913	WO 2001-US2356	20010125
	W: AU, BR, CA, CN, CZ, HU, IL, IN, JP, KR, MX, NZ, PL, RU, TR, ZA RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
EP	1265683	A1	20021218	EP 2001-908680	20010125
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
BR	2001008855	A	20030429	BR 2001-8855	20010125
JP	2003525730	T2	20030902	JP 2001-564860	20010125
US	2001036898	A1	20011101	US 2001-879031	20010612
US	6610624	B2	20030826		
ZA	2002006989	A	20030407	ZA 2002-6989	20020830
US	2004014590	A1	20040122	US 2003-620398	20030716
PRAI	US 1997-62099P	P	19971014		
	US 1998-157267	A2	19980921		
	US 2000-517883	A	20000303		
	WO 2001-US2356	W	20010125		
	US 2001-879031	A3	20010612		

AB Methods for removing soluble material from confined spaces within substrates such as containers, capsules and porous powders comprising extraction with supercrit. fluids, the pressure of which is preferably modulated between an upper level and a lower level within a relatively narrow range of fluid pressure and d. The method permits enhanced extraction efficiency, catalytic reaction rates and ability to maintain catalyst activity. A small amount of polyethylene glycol with an average mol. weight of 200 was pipetted into a 1-mL capped vial and the cap was pierced with a 500 mm needle. The level of the polymer was about 1/4" above the bottom of the vial. The polymer was then extracted at either a constant pressure of 165 bar or using the pressure modulation technique in the range of 159-172 bar. Temperature and extraction time

were 35° and 58 min resp. in both runs. Despite small pressure and d. modulation, the modulation technique was substantially more efficient at removing PEG 200 from the capped vial than conventional SFE. Extraction efficiency was nearly 7-fold higher than that of conventional SFE. The ability to rapidly modulate pressure appears to allow for very high extraction efficiency when compared to conventional SFE.

- ST extn reaction supercrit fluid  
 IT Drug delivery systems  
     (capsules; method for extraction and reaction using supercrit. fluids)  
 IT Density  
     Extraction  
     Lubricants  
     Nutrients  
     Plasticizers  
     Pressure  
     Supercritical fluids  
     Temperature  
         (method for extraction and reaction using supercrit. fluids)  
 IT Polyoxyalkylenes, uses  
     RL: NUU (Other use, unclassified); USES (Uses)  
         (method for extraction and reaction using supercrit. fluids)  
 IT 64-17-5, Ethanol, uses 124-38-9, carbon dioxide, uses 25322-68-3, polyethylene glycol  
     RL: NUU (Other use, unclassified); USES (Uses)  
         (method for extraction and reaction using supercrit. fluids)  
 IT 9005-25-8, starch, biological studies 22254-24-6, ipratropium bromide  
     RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses)  
         (method for extraction and reaction using supercrit. fluids)
- RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD
- RE
- (1) Anon; WO 9918939 1999 HCPLUS
  - (2) Anon; WO 9949996 1999
  - (3) Clark; US 5641510 1997 HCPLUS
  - (4) Deweese; US 5267455 1993
  - (5) Donsi; Pharm Acta helv 1991, V66(5-6), P170 HCPLUS
  - (6) Francis, A; J Phys Chem 1954, V58, P1099 HCPLUS
  - (7) Gallagher; US 5389263 1995 HCPLUS
  - (8) Gallagher; Am Chem Soc, Ch 22 1989, P334 HCPLUS
  - (9) Hannay; Royal Society of London Proceedings 1879, V29, P324
  - (10) Heit; US 5287632 1994
  - (11) Krukonis; US 5360478 1994 HCPLUS
  - (12) Larson; Biotechnology, Progress 1986, V2(2), P73 HCPLUS
  - (13) McHugh; Supercritical Fluid Extraction, Principles and Practice, 2nd Ed 1994, P369
  - (14) Modell; US 4061566 1977
  - (15) Modell; US 4338199 1982 HCPLUS
  - (16) Mohamed; AIChE Journal 1989, V35(2), P325 HCPLUS
  - (17) Pearson; US 4059308 1977
  - (18) Pearson; US 4163580 1979
  - (19) Sievers; US 4970093 1990 HCPLUS
  - (20) Subramaniam; US 5725756 1998 HCPLUS
  - (21) Subramaniam; US 5833891 1998 HCPLUS
  - (22) Tiltsher; US 4721826 1988 HCPLUS
  - (23) Tiltsher; Angew Chem Int Ed Engl 1981, V20, P892
  - (24) Tom; J Aerosol Sci 1991, V22(5), P555 HCPLUS
  - (25) Wetmore; US 5514220 1996
  - (26) Whitlock; US 5599381 1997 HCPLUS
  - (27) Yearout; US 3594983 1971
  - (28) Yeo; Biotechnology and Bioengineering 1993, V41, P341 HCPLUS
  - (29) Zosel; US 3806619 1974 HCPLUS
- IT 124-38-9, carbon dioxide, uses  
     RL: NUU (Other use, unclassified); USES (Uses)  
         (method for extraction and reaction using supercrit. fluids)

RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 13 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 2001:676650 HCAPLUS  
 DN 135:244446  
 ED Entered STN: 14 Sep 2001  
 TI Material processing by repeated solvent expansion-contraction  
 IN Saim, Said; Horhota, Stephen; Bochniak, David Joseph  
 PA Boehringer Ingelheim Pharmaceuticals, Inc., USA  
 SO PCT Int. Appl., 48 pp.  
 CODEN: PIXXD2  
 DT Patent  
 LA English  
 IC ICM B01D011-02  
 ICS B01D009-00  
 CC 48-1 (Unit Operations and Processes)  
 Section cross-reference(s): 63

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001066215	A1	20010913	WO 2001-US3019	20010130
	W: AU, BR, CA, CN, CZ, HU, IL, IN, JP, KR, MX, NZ, PL, RU, TR, US, ZA RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
	US 2001055561	A1	20011227	US 2001-774232	20010130
	EP 1263516	A1	20021211	EP 2001-906792	20010130
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
	BR 2001008912	A	20021224	BR 2001-8912	20010130
	JP 2003525731	T2	20030902	JP 2001-564861	20010130
	ZA 2002006943	A	20030404	ZA 2002-6943	20020829
PRAI	US 2000-186888P	P	20000303		
	WO 2001-US3019	W	20010130		

AB A method is disclosed for repeatedly converting a solvent from a state of solvent to a state of antisolvent with relatively little loss of solvent. The method is used to allow for processing of large amts. of solute material with min. amts. of solvent.

ST solute material processing solvent expansion contraction

IT Solvents  
 (antisolvents; material processing by repeated solvent expansion-contraction)

IT Coating process  
 Contraction (mechanical)  
 Crystallization

Drugs

Expansion  
 Extraction

Recycling

Solutes

Solvents

(material processing by repeated solvent expansion-contraction)

IT 64-17-5, Ethanol, uses 67-68-5, Dmso, uses 103-90-2, Acetaminophen

**124-38-9, Carbon dioxide, uses**

RL: TEM (Technical or engineered material use); USES (Uses)  
 (material processing by repeated **solvent expansion-contraction**)

RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Bisrat, M; WO 9852544 A 1998 HCPLUS
- (2) Cf Tech; EP 0868942 A 1998 HCPLUS
- (3) Davis, T; US 4536283 A 1985 HCPLUS
- (4) Robertson, J; WO 9858722 A 1998 HCPLUS
- (5) Upjohn Co; WO 9003782 A 1990 HCPLUS

IT **124-38-9, Carbon dioxide, uses**

RL: TEM (Technical or engineered material use); USES (Uses)  
 (material processing by repeated **solvent expansion-contraction**)

RN 124-38-9 HCPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 14 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN

AN 2001:676649 HCPLUS

DN 135:231760

ED Entered STN: 14 Sep 2001

TI Methods for extraction of drugs and related materials using supercritical fluids

IN Horhota, Stephen T.; Saim, Said

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO PCT Int. Appl., 54 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM B01D011-02

CC 63-8 (Pharmaceuticals)

Section cross-reference(s): 17, 48

FAN.CNT 3

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 2001066214	A1	20010913	WO 2001-US2356	20010125
	W: AU, BR, CA, CN, CZ, HU, IL, IN, JP, KR, MX, NZ, PL, RU, TR, ZA RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
→	US 6294194	B1	20010925	US 2000-517883	20000303
	EP 1265683	A1	20021218	EP 2001-908680	20010125
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR				
	BR 2001008855	A	20030429	BR 2001-8855	20010125
	JP 2003525730	T2	20030902	JP 2001-564860	20010125
PRAI	US 2000-517883	A	20000303		
→	US 1997-62099P	P	19971014		
	US 1998-157267	A2	19980921		
	WO 2001-US2356	W	20010125		
AB	Methods for removing soluble material from confined spaces within substrates such as containers, capsules and porous powders comprise extraction with supercrit. fluids, the pressure of which is preferably modulated between an upper level and a lower level within a relatively narrow range of fluid pressure and d. The method permits enhanced extraction efficiency, catalytic reaction rates and ability to maintain catalyst activity. The capsules containing a drug were extracted by using CO <sub>2</sub> at 65° at 552 bar.				
ST	supercrit fluid extn drug container				

IT Drug delivery systems  
 (capsules; extraction of drugs and related materials using supercrit. fluids)  
 IT Absorbents  
 Adsorbents  
 Catalysts  
 Containers  
 Drugs  
 Extraction apparatus  
 Lubricants  
 Plasticizers  
 Vials  
 (extraction of drugs and related materials using supercrit. fluids)  
 IT Polyoxyalkylenes, processes  
 RL: PEP (Physical, engineering or chemical process); PROC (Process)  
 (extraction of drugs and related materials using supercrit. fluids)  
 IT Extraction  
 (supercrit.; extraction of drugs and related materials using supercrit. fluids)  
 IT 64-17-5, Ethanol, processes 25322-68-3, Polyethylene glycol  
 RL: PEP (Physical, engineering or chemical process); PROC (Process)  
 (extraction of drugs and related materials using supercrit. fluids)  
 IT 22254-24-6, Ipratropium bromide  
 RL: PEP (Physical, engineering or chemical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)  
 (extraction of drugs and related materials using supercrit. fluids)  
 IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
 (supercrit.; extraction of drugs and related materials using supercrit. fluids)

RE.CNT 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Barton, J; WO 9949996 A 1999
- (2) Boehringer Ingelheim Pharma; WO 9918939 A 1999 HCPLUS
- (3) Krukonis, V; US 5514220 A 1996
- (4) Saim, S; US 5725756 A 1998 HCPLUS

IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
 (supercrit.; extraction of drugs and related materials using supercrit. fluids)

RN 124-38-9 HCPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 15 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2001:664895 HCPLUS  
 DN 135:262853  
 ED Entered STN: 12 Sep 2001  
 TI Predicting solubility in **supercritical solvents** using estimated virial coefficients and fluctuation theory  
 AU Tomberli, B.; Goldman, S.; Gray, C. G.  
 CS University of Guelph, Guelph-Waterloo Physics Institute, Guelph, ON, N1G 2W1, Can.  
 SO Fluid Phase Equilibria (2001), 187-188, 111-130  
 CODEN: FPEQDT; ISSN: 0378-3812  
 PB Elsevier Science B.V.  
 DT Journal

- LA English  
 CC 68-1 (Phase Equilibria, Chemical Equilibria, and Solutions)  
 Section cross-reference(s): 65, 69
- AB A theor. method based on combining the virial **expansion** and fluctuation theory for calculating the chemical potential of a **solute** in a **supercrit. fluid** is presented. The method is compared to literature results from Monte Carlo simulations based the Widom method for evaluating the chemical potential. For one-center and two-center Lennard Jones (2CLJ) potential models, the average difference from simulated results for the chemical potential is about 5 at densities up to twice the critical d. The method requires virial coeffs. up to C(T) (the third) to achieve this level of accuracy. Correlations based on corresponding states principles for the prediction of B(T) [AIChE J. 20 (1974) 263; AIChE J. 21 (1975) 827; AIChE J. 24 (1978) 1978] and C(T) [AIChE J. 29 (1983) 107] are used to estimate these virial coeffs. A comparison with exptl. determined values for naphthalene in **carbon dioxide** shows the ests. to be accurate at typical **supercrit. extraction** conditions. These correlations are then used to determine virial coeffs. and chemical potentials for naphthalene, benzoic acid and phenanthrene in **carbon dioxide** at several different state conditions for which solubility data exist. The theor. results are compared to chemical potentials obtained from exptl. solubility data.
- The method is found to be accurate, tractable and systematically improvable through the inclusion of higher order terms in the virial **expansion**.
- ST **solute supercrit solvent** solv virial coeff  
 fluctuation theory
- IT Statistical mechanics  
 (fluctuation theory; **solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)
- IT Chemical potential  
 Critical density  
 Lennard-Jones potential  
 Pair potential  
 Solubility  
 Solutes  
 Virial coefficient  
 (**solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)
- IT Extraction  
 Solvents  
 (**supercrit.**; **solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)
- IT 65-85-0, Benzoic acid, properties 85-01-8, Phenanthrene, properties 91-20-3, Naphthalene, properties 124-38-9, **Carbon dioxide**, properties  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)  
 (**solute** solubility in **supercrit. solvents** from estimated virial coeffs. and fluctuation theory)
- RE.CNT 45 THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD  
 RE
- (1) Anon; CRC Handbook of Chemistry and Physics, 73rd Edition 1993
  - (2) Anon; Design Institute for Physical Property Data American Institute of Chemical Engineers, Physical and Thermodynamic Properties of Pure Chemicals 1989
  - (3) Barker, J; J Chem Phys 1966, V44, P4206 HCPLUS
  - (4) Bartle, K; J Phys Chem Ref Data 1991, V20, P713 HCPLUS
  - (5) Bohm, H; Mol Phys 1984, V53, P651
  - (6) Bruno, T; Supercritical Fluid Technology 1991
  - (7) Buff, F; J Chem Phys 1955, V23, P458 HCPLUS
  - (8) Chialvo, A; AIChE J 1994, V40, P1558 HCPLUS

- (9) Ding, K; J Chem Phys 1993, V98, P3306 HCPLUS  
 (10) Dymond, J; The Virial Coefficients of Pure Gases and Mixtures: A Critical Compilation 1980  
 (11) Ely, J; J Chem Thermo 1989, V21, P879 HCPLUS  
 (12) Ewald, A; Faraday Soc Dis 1953, V15, P238  
 (13) Gray, C; Theory of Molecular Fluids 1984  
 (14) Guigard, S; Ind Eng Chem Res 1998, V37, P3786 HCPLUS  
 (15) Harvey, A; Fluid Phase Equilibria 1997, V130, P87 HCPLUS  
 (16) Jonah, D; Molecular-Based Study of Fluids, Advances in Chemistry Series 1983, V204 HCPLUS  
 (17) Joslin, C; Mol Phys 1996, V89, P489 HCPLUS  
 (18) Kirkwood, J; J Chem Phys 1951, V19, P774 HCPLUS  
 (19) Krishnan, T; Can J Chem 1979, V57, P530 HCPLUS  
 (20) Kurnik, R; J Chem Eng Data 1981, V26, P47 HCPLUS  
 (21) Kwak, T; Chem Eng Sci 1986, V41, P1303 HCPLUS  
 (22) Lamb, D; J Phys Chem 1986, V90, P4210 HCPLUS  
 (23) Lee, L; Supercritical Fluid Technology: Reviews in Modern Theory and Applications 1991  
 (24) Mason, E; The Virial Equation of State, 1st Edition 1969  
 (25) McQuarrie, D; Statistical Mechanics 1976  
 (26) Nouacer, M; Mol Sim 1989, V2, P55  
 (27) Orbey, H; AIChE J 1983, V29, P107 HCPLUS  
 (28) O'Connell, J; Mol Phys 1971, V20, P27  
 (29) Panagiotopoulos, A; Mol Sim 1992, V9, P1  
 (30) Prausnitz, J; Molecular Thermodynamics of Fluid-Phase Equilibria, 2nd Edition 1986  
 (31) Prigogine, I; The Molecular Theory of Solutions 1957  
 (32) Quiram, D; J Supercritical Fluids 1994, V7, P159 HCPLUS  
 (33) Reid, R; The Properties of Gases and Liquids, 4th Edition 1987  
 (34) Romano, S; Mol Phys 1979, V37, P17565  
 (35) Schmitt, W; J Chem Eng Data 1986, V31, P204 HCPLUS  
 (36) Shing, K; Mol Phys 1982, V46, P1109 HCPLUS  
 (37) Shing, K; Mol Phys 1988, V65, P1235 HCPLUS  
 (38) Snyder, J; J Chromatogr Sci 1993, V31, P183 HCPLUS  
 (39) Tomberli, B; PhD Thesis, University of Guelph 1998  
 (40) Tsonopoulos, C; AIChE 1975, V21, P827  
 (41) Tsonopoulos, C; AIChE 1978, V24, P1112  
 (42) Tsonopoulos, C; AIChE J 1974, V20, P263  
 (43) Vitzthum, O; DE 2357590 1975 HCPLUS  
 (44) Widom, B; J Chem Phys 1963, V39, P2808 HCPLUS  
 (45) Wojick, M; Mol Phys 1982, V45, P1209

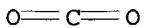
IT 124-38-9, Carbon dioxide, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties);  
 PROC (Process)

(solute solubility in supercrit. solvents from  
 estimated virial coeffs. and fluctuation theory)

RN 124-38-9 HCPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L98 ANSWER 16 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2001:107664 HCPLUS  
 DN 134:285514  
 ED Entered STN: 13 Feb 2001  
 TI Green process concepts for the pharmaceutical industry  
 AU Subramaniam, Bala; Saim, Said; Rajewski, Roger; Stella,  
 Valentino J.  
 CS Department of Chemical and Petroleum Engineering, University of Kansas,  
 Lawrence, KS, 66045-2223, USA

- SO ACS Symposium Series (2001), 766(Green Engineering), 96-110  
 CODEN: ACSMC8; ISSN: 0097-6156
- PB American Chemical Society
- DT Journal; General Review
- LA English
- CC 63-0 (Pharmaceuticals)
- AB A review with 23 refs. Process concepts for producing drug particles using **supercrit. carbon dioxide** (scCO<sub>2</sub>) as an **antisolvent** and for substrate **coating** employing scCO<sub>2</sub> as the fluidizing medium and **antisolvent** are described. Particle micronization with scCO<sub>2</sub> allows for reproducible **crystal** formation with the potential for increased surface area and dissoln. rates. **Coating** with scCO<sub>2</sub> allows the use of traditional organic-soluble **coatings** with complete **solvent** recovery and virtually no atmospheric emissions. For formation of drug nanoparticles, an ultrasonic nozzle that employs scCO<sub>2</sub> as the energizing medium is used to form droplets of the drug-laden solution. The scCO<sub>2</sub> also selectively **exts.** the **solvent** from the droplets, **precipitating** the drug. Submicron particles of hydrocortisone and ibuprofen (600 nm or less) formed in this manner are presented. Advantages include the production of virtually **solvent-free** drug particles in a narrow size range. For particle **coating**, scCO<sub>2</sub> is used to fluidize the core substrate particles. The scCO<sub>2</sub> also removes the **solvent** from the **coating** solution sprayed on the substrates, thereby **precipitating** the **coating**. This **coating** process **expands** the range of substrate/coating combinations possible with the conventional air-suspension Wurster **coater**, making it feasible to coat water-soluble substrates with **solutes** sprayed from organic solns.
- ST review pharmaceutical industry green process
- IT Pharmaceutical industry  
 (green process concepts for pharmaceutical industry)
- IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
 (supercrit.; green process concepts for pharmaceutical industry)
- RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD
- RE
- (1) Anastas, P; Green Chemistry: Frontiers in Benign Chemical Syntheses and Processes 1998
  - (2) Borel, J; Ann NY Acad Sci 1985, V475, P307
  - (3) Dixon, D; AIChE J 1993, V39, P127 HCAPLUS
  - (4) Dixon, D; AIChE J 1993, V39, P127 HCAPLUS
  - (5) Eggers, R; High Pressure Chemical Engineering; Process Technology Proceedings 1996, V12, P247 HCAPLUS
  - (6) Ensminger, D; Ultrasonics: Fundamentals, Technology, Applications, 2nd ed 1988
  - (7) Gallagher, P; ACS Symposium Series 1989, 406, P334
  - (8) Hartmann, J; Journal of Scientific Instruments 1939, V16, P140
  - (9) Kreuter, J; J Controlled Release 1989, V16, P169
  - (10) Matson, D; Ind Eng Chem Res 1987, V26, P2298 HCAPLUS
  - (11) Mawson, S; J Appl Polym Sci 1997, V64, P2105 HCAPLUS
  - (12) Muller, B; German Patent Appl No DE 3744329 A1 1989
  - (13) Randolph, T; Biotechnol Prog 1993, V9, P429 HCAPLUS
  - (14) Saim, S; Pharm Res 1996, V13, PS-273
  - (15) Sanchez, A; Int J Pharm 1993, V99, P263 HCAPLUS
  - (16) Span, R; J Phys Chem Ref Data 1996, V25, P1509 HCAPLUS
  - (17) Subramaniam, B; US 5833891 1998 HCAPLUS
  - (18) Subramaniam, B; J Pharm Sci 1997, V86, P885 HCAPLUS
  - (19) Tom, J; Biotechnol Prog 1991, V7, P403 HCAPLUS
  - (20) Tomlinson, E; Int J Pharm 1983, V4, P49 HCAPLUS
  - (21) Wurster, D; J Amer Pharm Assoc Sci Ed 1959, V48, P451 MEDLINE

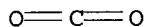
- (22) Yeo, S; Biotech Bioeng 1993, V41, P341 HCAPLUS  
 (23) York, P; Respiratory Drug Delivery V: Program and Proceedings 1996, P231

HCAPLUS

IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
 (supercrit.; green process concepts for pharmaceutical industry)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L98 ANSWER 17 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1999:806362 HCAPLUS  
 DN 132:109880  
 ED Entered STN: 22 Dec 1999  
 TI Supercritical fluid extraction of solids.  
 Statistical thermodynamic approach  
 AU Boublík, Tomas  
 CS Department of Physical and Macromolecular Chemistry, Charles University,  
 Faculty of Science, Prague, Czech Rep.  
 SO Physical Chemistry Chemical Physics (2000), 2(1), 91-95 ←  
 CODEN: PPCPFQ; ISSN: 1463-9076  
 PB Royal Society of Chemistry  
 DT Journal  
 LA English  
 CC 48-1 (Unit Operations and Processes)  
 Section cross-reference(s): 69  
 AB To study the effect of non-sphericity of solvent and  
 solute mols. on the main characteristics of supercrit.  
 fluid extraction the fourth-order virial expansion  
 is considered in which the individual virial coeffs. (and cross terms) are  
 determined from the formula proposed recently for the Kihara generalized pair  
 potential. The Kihara four-step square-well potential is assumed; its  
 form makes it possible to write analytic expressions for the considered  
 virial coeffs. and, consequently, for the main thermodn. functions - the  
 residual chemical potential of solute and total pressure. The  
 method is applied to determine the dependence of the mole fraction of  
 solute on temperature or pressure in the binary systems carbon  
 dioxide-naphthalene and ethylene-naphthalene and the effect of the  
 cosolvent on the solute concentration in the system  
 ethylene-naphthalene-acetone at 308 K. Fair agreement with the simulation  
 and exptl. data was found.  
 ST supercrit fluid extn solid statistical  
 thermodn analysis; carbon dioxide naphthalene  
 supercrit fluid extn; ethylene naphthalene  
 supercrit fluid extn  
 IT Solids  
 Statistical thermodynamics  
 (statistical thermodn. approach in supercrit. fluid  
 extraction of solids)  
 IT Extraction  
 (supercrit.; statistical thermodn. approach in  
 supercrit. fluid extraction of solids)  
 IT 124-38-9, Carbon dioxide, processes  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties); TEM  
 (Technical or engineered material use); PROC (Process); USES (Uses)  
 (statistical thermodn. approach in supercrit. fluid  
 extraction of solids)

IT 67-64-1P, Acetone, processes 74-85-1P, Ethylene, processes 91-20-3P,  
 Naphthalene, processes  
 RL: PUR (Purification or recovery); REM (Removal or disposal); PREP  
 (Preparation); PROC (Process)  
 (statistical thermodyn. approach in **supercrit. fluid**  
**extraction of solids**)

RE.CNT 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Barker, J; J Chem Phys 1962, V36, P2558 HCPLUS
- (2) Barker, J; J Chem Phys 1962, V36, P2564 HCPLUS
- (3) Boublik, T; J Chem Phys 1987, V87, P1751 HCPLUS
- (4) Boublik, T; Mol Phys 1981, V42, P209 HCPLUS
- (5) Boublik, T; Mol Phys 1997, V90, P585 HCPLUS
- (6) Chen, S; Ber Bunsen-Ges Phys Chem 1977, V81, P1048 HCPLUS
- (7) Deiters, U; Ber Bunsen-Ges Phys Chem 1984, V88, P791 HCPLUS
- (8) Goldman, S; J Phys Chem 1996, V100, P7246 HCPLUS
- (9) Hirschfelder, J; Molecular Theory of Gases and Liquids 1954
- (10) Kihara, T; Adv Chem Phys 1963, V5, P147 HCPLUS
- (11) McHugh, M; Supercritical Fluid Extraction 1994
- (12) Pavlicek, J; J Phys Chem 1992, V98, P2298
- (13) Sindelka, M; Mol Phys 1999, V97, P1035 HCPLUS
- (14) Sindelka, M; Mol Phys 1999, V96, P243 HCPLUS
- (15) Suoqi, Z; J Supercrit Fluids 1995, V8, P15
- (16) Tsekhaneskaya, Y; Zh Fiz Khim 1964, V38, P2106

IT 124-38-9, Carbon dioxide, processes

RL: PEP (Physical, engineering or chemical process); PRP (Properties); TEM  
 (Technical or engineered material use); PROC (Process); USES (Uses)  
 (statistical thermodyn. approach in **supercrit. fluid**  
**extraction of solids**)

RN 124-38-9 HCPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L98 ANSWER 18 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN

AN 1999:262169 HCPLUS

DN 130:301697

ED Entered STN: 29 Apr 1999

TI Methods of treating capsules and dry, powdered pharmaceutical formulations

IN Horhota, Steven T.; Said, Saim

PA Boehringer Ingelheim Pharmaceuticals, Inc., USA

SO PCT Int. Appl., 79 pp.

CODEN: PIXXD2

DT Patent

LA English

IC ICM A61K009-48

ICS A61K009-14

CC 63-6 (Pharmaceuticals)

FAN.CNT 3

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9918939	A1	19990422	WO 1998-US20815	19981005
	W:	AU, BG, BR, CA, CN, CZ, EE, HU, IL, JP, KR, LT, LV, MX, NZ, PL, RO, RU, SI, TR			
	RW:	AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE			
	US 6228394	B1	20010508	US 1998-157267	19980921
	CA 2302276	AA	19990422	CA 1998-2302276	19981005
	AU 9897838	A1	19990503	AU 1998-97838	19981005
	AU 753076	B2	20021010		

EP 1024794	A1	20000809	EP 1998-952043	19981005
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
BR 9814818	A	20001003	BR 1998-14818	19981005
EE 200000292	A	20010815	EE 2000-200000292	19981005
JP 2001519380	T2	20011023	JP 2000-515575	19981005
NZ 504394	A	20021220	NZ 1998-504394	19981005
ZA 9809261	A	19990531	ZA 1998-9261	19981012
MX 200003329	A	20001110	MX 2000-3329	20000405
BG 104317	A	20001229	BG 2000-104317	20000407
PRAI US 1997-62099P	P	19971014		
US 1998-157267	A	19980921		
WO 1998-US20815	W	19981005		
<b>AB</b>	Undesirable materials present in gelatin, cellulose or plastic capsules used for storing a dry, powdered pharmaceutical formulation are extracted by supercrit. fluids. The method is also used for removing undesirable material from drug powder. The amount of powder retained in the capsules following inhalation is minimized. A powder blend of lactose and ipratropium bromide was loaded into CO <sub>2</sub> -treated capsules and significant reduction in the amts. of drug or carrier retained in the capsules following inhalation was demonstrated.			
<b>ST</b>	supercrit fluid extn capsule impurity removal; ipratropium bromide lactose powder inhalant			
<b>IT</b>	Drug delivery systems (capsules; removal of impurities from capsules containing powdery ingredients by supercrit. fluid extraction)			
<b>IT</b>	Drug delivery systems (inhalants; removal of impurities from capsules containing powdery ingredients by supercrit. fluid extraction)			
<b>IT</b>	Lubricants			
	Water vapor (removal of impurities from capsules containing powdery ingredients by supercrit. fluid extraction)			
<b>IT</b>	Extraction (supercrit.; removal of impurities from capsules containing powdery ingredients by supercrit. fluid extraction)			
<b>IT</b>	<b>124-38-9, Carbon dioxide, uses</b> RL: NUU (Other use, unclassified); USES (Uses) (removal of impurities from capsules containing powdery ingredients by supercrit. fluid extraction)			
<b>IT</b>	63-42-3, Lactose 22254-24-6, Ipratropium bromide RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses) (removal of impurities from capsules containing powdery ingredients by supercrit. fluid extraction)			
RE.CNT 6	THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD			
RE	(1) Genentech Inc; US 5641510 A HCAPLUS (2) Genentech Inc; WO 9601105 A 1996 HCAPLUS (3) Heit; US 5287632 A 1994 (4) Minnesota Mining And Manufacturing Company; WO 9518834 A 1995 HCAPLUS (5) Sumitomo Heavy Industries; DE 3545913 A 1986 HCAPLUS (6) Syntex U S A Inc; EP 0421577 A 1991 HCAPLUS			
<b>IT</b>	<b>124-38-9, Carbon dioxide, uses</b> RL: NUU (Other use, unclassified); USES (Uses) (removal of impurities from capsules containing powdery ingredients by supercrit. fluid extraction)			
RN	124-38-9 HCAPLUS			
CN	Carbon dioxide (8CI, 9CI) (CA INDEX NAME)			

L98 ANSWER 19 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1998:731765 HCAPLUS  
 DN 129:347344  
 ED Entered STN: 18 Nov 1998  
 TI Methods for a particle precipitation and coating using near-critical and supercritical antisolvents  
 IN Subramaniam, Bala; Saim, Said; Rajewski, Roger A.; Stella, Valentino  
 PA The University of Kansas, USA  
 SO U.S., 30 pp., Cont.-in-part of U.S. Ser. No. 722,463.  
 CODEN: USXXAM  
 DT Patent  
 LA English  
 IC ICM B01B011-00  
 ICS B01J002-04; B05D001-02  
 NCL 264007000  
 CC 63-8 (Pharmaceuticals)  
 FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 5833891	A	19981110	US 1997-805215	19970227
	US 5874029	A	19990223	US 1996-723463	19961009
	CA 2247900	AA	19970904	CA 1997-2247900	19970228
	WO 9731691	A1	19970904	WO 1997-US3207	19970228
	W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
	AU 9721936	A1	19970916	AU 1997-21936	19970228
	AU 709384	B2	19990826		
	EP 885038	A2	19981223	EP 1997-914827	19970228
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
PRAI	JP 2002504011	T2	20020205	JP 1997-531174	19970228
	US 1996-723463	A2	19961009		
	US 1996-12592P	P	19960301		
	US 1996-12593P	P	19960301		
	US 1997-805215	A	19970227		
	WO 1997-US3207	W	19970228		

AB Improved methods and apparatus for particle precipitation and coating using near- or

supercrit. fluid conditions are described. A fluid dispersion having a continuous phase dispersant and at least one precipitatable substance therein is contacted with a supercrit. fluid (SCF) antisolvent so as to generate focused high frequency antisolvent sonic waves, breaking up the dispersion into extremely small droplets; the enhanced mass transfer rates between the droplets and the antisolvent causes precipitation of very small particles on the order of 0.1-10 µm. In coating processes, a turbulent fluidized flow of core particles is created using an SCF antisolvent in an enclosed zone. The core particles are contacted therein at near- or supercrit. conditions by a fluid dispersion containing a dispersant together with a precipitatable substance. The antisolvent depletes the dispersant and the substance is precipitated onto the fluidized core particles. In another

aspect of the invention, a process for preparing and administering a medicament using only a single container is provided. In such method, a fluid dispersion having a dispersant with the medicament therein is contacted with an antisolvent at near- or supercrit. conditions within a

in use container, so as to directly precipitate small particles of the medicament

the container. The antisolvent is then removed and the use container is sealed with the medicament particles therein. Thereafter, dose(s) of the medicament can be withdrawn from the use container and administered to a patient. Examples are given for recrystn. of hydrocortisone , RG503H, ibuprofen, or camptothecin from a DMSO solution using compressed CO<sub>2</sub> as energizing gas and antisolvent.

ST recrystn drug particle pptn supercrit antisolvent

IT Solvents

(antisolvents; particle precipitation and coating using near-critical and supercrit. antisolvents)

IT Electromagnetic wave

(high-frequency; particle precipitation and coating using near-critical and supercrit. antisolvents)

IT Coating materials

Disperse systems

Dispersing agents

Particle size

Particles

Recrystallization

Supercritical fluids

(particle precipitation and coating using near-critical and supercrit. antisolvents)

IT 74-98-6, Propane, properties 75-28-5, Isobutane 75-46-7,

Trifluoromethane 106-97-8, Butane, properties 124-38-9,

Carbon dioxide, properties 2551-62-4, Sulfur

hexafluoride 10024-97-2, Nitrous oxide, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties);

PROC (Process)

(antisolvent and energizing gas; particle precipitation and coating using near-critical and supercrit. antisolvents)

IT 7440-59-7, Helium, properties 7727-37-9, Nitrogen, properties

7782-44-7, Oxygen, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties);

PROC (Process)

(energizing gas; particle precipitation and coating using near-critical and supercrit. antisolvents)

IT 50-23-7, Hydrocortisone 67-68-5, Dmso, properties 7689-03-4,

Camptothecin 15687-27-1, Ibuprofen 34346-01-5, Glycolic acid-lactic acid copolymer

RL: PEP (Physical, engineering or chemical process); PRP (Properties);

PROC (Process)

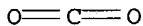
(particle precipitation and coating using near-critical and supercrit. antisolvents)

RE.CNT 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

- (1) Anon; WO 9201446 1992 HCPLUS
- (2) Anon; EP 0542314 1993 HCPLUS
- (3) Anon; WO 9501221 1995
- (4) Anon; WO 9501324 1995 HCPLUS
- (5) Anon; Heat Systems Ultrasonics, Inc Brochure, Sonimist Ultrasonic Spray Nozzles
- (6) Barry; US 4900558 1990 HCPLUS
- (7) Bodmeier; Pharmaceutical Research 1995, V12(8) HCPLUS
- (8) Debenedetti; NATO ASI Series, E: Applied Sciences 1994, V273
- (9) Dixon; AIChE Journal 1993, V39(1), P127 HCPLUS
- (10) Dixon; J Applied Polymer Science 1993, V50, P1929 HCPLUS
- (11) Dixon; Polymer 1994, V35(18) HCPLUS
- (12) Fischer; US 5043280 1991 HCPLUS
- (13) Gallagher; US 5389263 1995 HCPLUS
- (14) Kim; US 5344676 1994
- (15) Kurkonis; US 5360478 1994 HCPLUS

- (16) Lefebvre; Atomization and Sprays 1989, P136  
 (17) Niwa; Journal of Controlled Release 1993, V25, P89 HCAPLUS  
 (18) Prince; US 5308648 1994 HCAPLUS  
 (19) Randolph; Biotechnol Prog 1993, V9(4) HCAPLUS  
 (20) Sanchez; International Journal of Pharmaceutics 1993, V99, P263 HCAPLUS  
 (21) Sievers; US 5301664 1994  
 (22) Tom; Biotechnol 1991, V7, P403 HCAPLUS  
 (23) Wilcox; A I Ch E Journal 1965, V11(1), P69 HCAPLUS  
 (24) Yeo; Biotechnology and Bioengineering 1993, V41, P341 HCAPLUS  
 (25) Yeo; J Pharmaceutical Sciences 1994, V83(12) HCAPLUS  
 (26) Yeo; Macromolecules 1993, V26, P6207 HCAPLUS  
 (27) York; Respiratory Drug Delivery V 1996, P231 HCAPLUS
- IT 124-38-9, Carbon dioxide, properties  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties);  
 PROC (Process)  
 (antisolvent and energizing gas; particle precipitation and coating using  
 near-critical and supercrit. antisolvents)
- RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



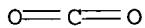
L98 ANSWER 20 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1998:140144 HCAPLUS  
 ED Entered STN: 09 Mar 1998  
 TI The behavior of micellar systems formed in **supercritical carbon dioxide** and their use as nanobioreactors.  
 AU Niemeyer, E. D.; Bonzagni, N. J.; Bright, F. V.  
 CS Department Chemistry, State University New York, Buffalo, NY, 14260-3000,  
 USA  
 SO Book of Abstracts, 215th ACS National Meeting, Dallas, March 29-April 2  
 (1998), PHYS-251 Publisher: American Chemical Society, Washington, D. C.  
 CODEN: 65QTAA  
 DT Conference; Meeting Abstract  
 LA English  
 AB It is well known that the physicochem. properties of **supercrit. fluids** (SFs) can be tuned between **gas** and liquid-like values with only slight changes in temperature and pressure. This tunability has helped to make SFs attractive **solvents** for use in chemical reactions, sepns., and **extraction** techniques. While **supercrit. CO<sub>2</sub>** (scCO<sub>2</sub>) is environmentally responsible, inexpensive, industrially applicable, and the most commonly used SF, it is a poor **solvent** for polar **solute**s. Reverse micelles offer a convenient methodol. to enhance the solubility of hydrophiles and expand the applicability of scCO<sub>2</sub>. Recently, we and others demonstrated that one can form stable reverse micelles in scCO<sub>2</sub> using a perfluoropolyether-based surfactant (PFPE) and host hydrophiles as large as proteins. This presentation will focus on our efforts to determine the characteristics of the interior water pool within these micelles and their use as nanobioreactors for enzyme catalysis.

L98 ANSWER 21 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1997:298833 HCAPLUS  
 DN 127:56433  
 ED Entered STN: 10 May 1997  
 TI On the suitability of the virial equation for modeling the solubility of solids in **supercritical fluids**  
 AU Harvey, Allan H.  
 CS Physical and Chemical Properties Division, Chemical Science and Technology Laboratory, National Institute of Standards and Technology, Boulder, CO,

USA  
 SO Fluid Phase Equilibria (1997), 130(1-2), 87-100  
 CODEN: FPEQDT; ISSN: 0378-3812  
 PB Elsevier  
 DT Journal  
 LA English  
 CC 68-1 (Phase Equilibria, Chemical Equilibria, and Solutions)  
 AB Five model systems, the van der Waals fluid, the Soave-Redlich-Kwong fluid, the Peng-Robinson fluid, the hard-sphere fluid, and the square-well fluid, are used to examine the performance of the truncated virial expansion in describing the fugacity of a solute at infinite dilution in a solvent. It is demonstrated that the virial fugacity results deteriorate at significantly lower densities as the solute becomes larger. This has consequences for attempts to describe the solubility of solids in supercrit. fluids, where the virial expansion, truncated after the third virial coefficient, has been considered as a modeling option. The results of this work suggest that, for the densities and solute-to-solvent size ratios commonly encountered in supercrit. extraction, the truncated virial expansion should not be expected to describe correctly the solute fugacity, and therefore any success it has in fitting solubility data should be viewed with caution.  
 ST solv solid supercrit fluid virial equation;  
 solute fugacity supercrit fluid virial  
 equation  
 IT Phase equilibrium  
     (fluid-solid; suitability of virial equation for modeling solubility of solids in supercrit. fluids)  
 IT Fugacity  
     Hard-sphere model  
     Peng-Robinson equation of state  
     Soave-Redlich-Kwong equation of state  
     Solubility  
     Square well potential  
     Van der Waals equation of state  
     Virial equation of state  
     (suitability of virial equation for modeling solubility of solids in supercrit. fluids)  
 IT Solvents  
     (supercrit.; suitability of virial equation for modeling solubility of solids in supercrit. fluids)  
 L98 ANSWER 22 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1995:615662 HCPLUS  
 DN 123:40911  
 ED Entered STN: 16 Jun 1995  
 TI Formation of fine powders of caffeine by RESS  
 AU Ksibi, H.; Subra, P.; Garrabos, Y.  
 CS Universite de Paris XIII, Villetteuse, 93430, Fr.  
 SO Advanced Powder Technology (1995), 6(1), 25-33  
 CODEN: APTEEE; ISSN: 0921-8831  
 PB VSP  
 DT Journal  
 LA English  
 CC 63-8 (Pharmaceuticals)  
 AB Precipitation of solids resulting from solution supersatn. is widely adopted to produce organic and inorg. powders. In fact, the rapid expansion of supercrit. solution (RESS) is a new process of particle formation. Various morphologies and particle sizes can be thus produced: thin films, thin diameter fibers, needles or spherical products of narrow size distribution. The distinguishing features of this process are the fast attainment of the uniform conditions and of high supersaturations

in the carrier fluid (**supercrit. carbon dioxide**), which favor the formation of small particles, with narrow distribution. The **expansion** of a **supercrit.** solution thus leads to loss of **solvent** power and hence to **solute precipitation**. The RESS is described for the production of fine powders of caffeine from **supercrit. carbon dioxide** upon **expansion**. There is variety of the fluid solution **expansion** parameters. The product morphol., however, can vary considerably depending on the solution components and the operating conditions used in the process: **solute** concentration, preexpansion and **expansion** temperature and pressure of **extraction** have been shown to affect the product characteristics of the formed powder during the process. Optical photomicrographs of the formed particles are compared taking into account the variation of thermodn. variables. Finally, the variation of the d. distribution and the particle sizes along a plate of deposition is discussed.

- ST caffeine powder **supercrit** carbon dioxide  
**expansion**
- IT Particle size  
 (production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)
- IT Pharmaceutical dosage forms  
 (powders, production of fine powders by rapid **expansion** of **supercrit. solns.**)
- IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)
- IT 58-08-2, Caffeine, processes  
 RL: PEP (Physical, engineering or chemical process); PROC (Process)  
 (production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)
- IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (production of fine powders of caffeine by rapid **expansion** of **supercrit. carbon dioxide**)
- RN 124-38-9 HCAPLUS
- CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



- L98 ANSWER 23 OF 27 HCAPLUS COPYRIGHT 2004 ACS on STN
- AN 1994:674375 HCAPLUS
- DN 121:274375
- ED Entered STN: 10 Dec 1994
- TI Solute trapping in off-line **supercritical** fluid **extraction** using controlled modifier condensation.
- AU Vejrosta, Jiri; Ansorgova, Alena; Planeta, Josef; Breen, David G.; Bartle, Keith D.; Clifford, Anthony A.
- CS Institute of Analytical Chemistry, Academy of Sciences of the Czech Republic, Veveri 97, Brno, 611 42, Czech.
- SO Journal of Chromatography, A (1994), 683(2), 407-10
- CODEN: JCRAEY; ISSN: 0021-9673
- PB Elsevier
- DT Journal
- LA English
- CC 5-1 (Agrochemical Bioregulators)
- Section cross-reference(s): 80
- AB A new approach to **solvent** trapping, based on controlled modifier condensation, is presented. The trapping system consists of a

fused-silica capillary equipped with a cryofocusing device. As a trapping mechanism, nebulization of **expanding supercrit.** mixture with condensing modifier, followed by analyte trapping into moving liquid layer is assumed. In spiking expts., flufenoxuron was **extracted** with 10% methanol-modified CO<sub>2</sub> and recoveries >90% were found. The resulting **solvent** vols. needed for quant. trapping are much lower (ca. 0.3 mL) than in the case of direct bubbling through bulk liquid

ST flufenoxuron analytical **supercrit fluid extn**

IT **Extraction**

(anal.; **solute** trapping in off-line **supercrit.**

**fluid extraction** using controlled modifier condensation)

IT 101463-69-8, Flufenoxuron

RL: ANT (Analyte); ANST (Analytical study)

(**solute** trapping in off-line anal. **supercrit.**

**fluid extraction** using controlled modifier condensation)

L98 ANSWER 24 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN

AN 1994:614317 HCPLUS

DN 121:214317

ED Entered STN: 29 Oct 1994

TI The entropy of hydration of simple hydrophobic **solutes**

AU Paulaitis, Michael E.; Ashbaugh, Henry S.; Garde, Shekhar

CS Center for Molecular and Engineering Thermodynamics, Department of Chemical Engineering, University of Delaware, Newark, DE, 19716, USA

SO Biophysical Chemistry (1994), 51(2-3), 349-57

CODEN: BICIAZ; ISSN: 0301-4622

DT Journal

LA English

CC 69-2 (Thermodynamics, Thermochemistry, and Thermal Properties)

AB Infinite-dilution partial molar entropies of solvation of simple, monoat.

**solutes** in water are defined in terms of the entropy associated with (1) **solute** insertion at constant volume and at a fixed position in the **solvent**, and (2) **expansion** or **contraction**

of the pure **solvent** to maintain constant pressure. A statistical mech. **expansion** for the entropy of solution in terms of

multiparticle correlation functions is applied to this definition to identify three intrinsic contributions to the hydration entropy -

**solute-solvent** pair correlations, rearrangement of **solvent** in the vicinity of the **solute** mol., and

**expansion** or **contraction** of the pure **solvent** -

which the authors evaluate for the inert **gases** in water at

25°C. For the smaller **solutes**, it was found that the

**solvent** reorganization and **solvent expansion**

contributions offset one another such that the entropy of hydration is

determined almost exclusively by **solute**-water pair correlations. The

**solute**-water pair correlation entropy also prevails as the primary

factor determining entropies of hydration for the larger **solutes**;

however, **solvent** reorganization now makes a small, neg.

contribution to the entropy.

ST partial molar entropy hydration hydrophobic **solute**;

**solvent** **solute** correlation hydration entropy calcn

IT Hydration, chemical

(statistical mech. calcn.; entropy of hydration of simple hydrophobic **solutes**)

IT **Solutes**

(**hydrophobic**, statistical mech. calcn.; entropy of hydration of simple **hydrophobic** **solutes**)

IT Distribution function

(pair correlation, **solute**-water; entropy of hydration of simple **hydrophobic** **solutes**)

IT Entropy

(partial molar, of hydration; entropy of hydration of simple **hydrophobic** **solutes**)

L98 ANSWER 25 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1988:552169 HCPLUS  
 DN 109:152169  
 ED Entered STN: 28 Oct 1988  
 TI **Supercritical fluid extraction of**  
 particulate and adsorbent materials. Part 2  
 AU Wright, B. W.; Smith, R. D.  
 CS Battelle Pac. Northwest Lab., Richland, WA, USA  
 SO Report (1987), EPA/600/4-87/040; Order No. PB88-133699, 80 pp. Avail.:  
 NTIS  
 From: Gov. Rep. Announce. Index (U. S.) 1988, 88(7), Abstr. No. 817,000  
 DT Report  
 LA English  
 CC 48-1 (Unit Operations and Processes)  
 Section cross-reference(s): 79, 80  
 AB The phys. properties of **supercrit.** fluids provide  
 similar **solvent** strengths as liqs. with higher diffusion  
 coeffs., lower viscosities, and an extended temperature range which provides  
 the potential for more rapid and efficient **extraction** rates. The report  
 describes **expanded** studies for evaluating the applicability and  
 efficiency of anal. **supercrit.-fluid extraction**  
 and related methodologies. These studies included the development of  
 quant., off-line, **supercrit.-fluid extraction**  
 methodol. and a comparison to traditional Soxhlet **extraction**, the  
 development and evaluation of online, **supercrit.-fluid**  
**extraction-gas chromatog.** for combined sample preparation and  
 anal., and direct **supercrit.-fluid extn**  
**-mass spectrometry** for the monitoring of specific **extraction**  
 profiles as a function of time. The sample matrixes included an air  
 particulate sample and XAD-2 resin, polyurethane foam, and Spherocarb  
 adsorbents that were spiked with various model compds. CO<sub>2</sub>,  
 isobutane, and MeOH-modified (20 mol %) CO<sub>2</sub> were used as  
**supercrit. fluids.** Related studies on the evaluation of  
 the quant. anal. capability of a fluorescence-detection, **supercrit**  
**-fluid chromatog.** method and the development of viable  
 solute focusing methods for capillary **supercrit.-**  
**fluid chromatog.** were conducted.  
 ST adsorbent **extn supercrit fluid;** analysis  
**extn supercrit fluid;** gas chromatog  
**extn supercrit fluid;** mass spectrometry  
**extn supercrit fluid**  
 IT Extraction  
 (by **supercrit. fluid**, of adsorbents and solids)  
 IT Chromatography, gas  
 Mass spectroscopy  
 (extraction by **supercrit. fluid** in combination  
 with)  
 IT Analysis  
 (extraction in, by **supercrit. fluid**)  
 IT Adsorbents  
 (extraction of, by **supercrit. fluid**)

L98 ANSWER 26 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1982:562235 HCPLUS  
 DN 97:162235  
 ED Entered STN: 12 May 1984  
 TI Chromatographic study of the thermodynamics of solutions of hydrocarbons  
 in liquid **crystal solvents.** Evidence for order  
 disturbance by the **solutes**  
 AU Klunder, H.; De Ligny, C. L.  
 CS Lab. Anal. Chem., Univ. Utrecht, 3522 AD, Neth.

- SO Journal of Solution Chemistry (1982), 11(3), 169-88  
 CODEN: JSLCAG; ISSN: 0095-9782
- DT Journal
- LA English
- CC 22-13 (Physical Organic Chemistry)
- AB Gas chromatog. expts. were carried out in various phases of the solvents 4-acetoxy-N-(4-methoxybenzylidene)aniline, dibutoxyazoxybenzene, Li stearate, dihexyloxyazoxybenzene, and diheptyloxyazoxybenzene. The solutes were linear, branched and cyclic alkanes, and substituted benzenes. Excess enthalpies, entropies, and free entropies were calculated from net retention vols. In the nematic liquid crystalline phases the effect of order disturbances was significant in H<sub>2</sub>e and S<sub>2</sub>e but it was, by enthalpy-entropy compensation, not demonstrable in .hivin.G<sub>2</sub>e. Differences in flexibility and degree of expansion of the solutes did not result in significantly different values of the excess quantities.
- ST chromatog solute liq crystal; heat mixing  
 solute liq crystal; orientation liq crystal  
 solute
- IT Heat of mixing  
 (of hydrocarbons with liquid crystal solvents,  
 chromatog. study of)
- IT Chromatography, gas  
 (of solutes in liquid crystals, order disturbance in  
 relation to)
- IT Liquid crystals  
 (orientation of, effect of solutes on, chromatog. in relation  
 to)
- IT Alkanes, properties  
 Hydrocarbons, properties  
 RL: PRP (Properties)  
 (thermodn. of solution with liquid crystal solvents,  
 chromatog. study of)
- IT Entropy  
 Free energy  
 (excess, of hydrocarbons with liquid crystal solvent,  
 chromatog. study of)
- IT 2587-42-0 2635-26-9 4485-12-5 10484-13-6 17051-01-3  
 RL: PRP (Properties)  
 (orientation of, effect of solutes on, gas  
 chromatog. in relation to)
- IT 540-84-1 541-73-1 560-21-4 563-16-6 583-48-2 589-43-5 590-73-8  
 592-27-8 609-26-7 619-99-8 624-29-3 638-04-0 1067-20-5  
 1071-26-7 2207-03-6 2207-04-7 2213-23-2 2216-33-3 3221-61-2  
 3522-94-9 4032-86-4 6876-23-9 15869-80-4 16747-26-5 95-47-6,  
 uses and miscellaneous 95-49-8 95-50-1 106-42-3, uses and  
 miscellaneous 106-43-4 106-46-7 108-38-3, uses and miscellaneous  
 108-41-8 111-65-9, uses and miscellaneous  
 RL: PROC (Process)  
 (solns. in liquid crystal solvents, chromatog. of)
- IT 98-06-6 104-51-8 111-84-2 124-18-5 493-01-6 493-02-7 620-14-4  
 622-96-8 871-83-0 922-28-1 926-82-9 1069-53-0 2207-01-4  
 2216-30-0 2216-34-4 15869-87-1  
 RL: PROC (Process)  
 (solns. with liquid crystal solvents, chromatog. of)
- L98 ANSWER 27 OF 27 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1979:175379 HCPLUS  
 DN 90:175379  
 ED Entered STN: 12 May 1984  
 TI Solid solubilities of heavy hydrocarbons in supercritical  
 solvents  
 AU Mackay, Michael E.; Paulaitis, Michael E.

CS Dep. Chem. Eng., Univ. Delaware, Newark, DE, USA  
 SO Industrial & Engineering Chemistry Fundamentals (1979), 18(2), 149-53  
 CODEN: IECFA7; ISSN: 0019-7874  
 DT Journal  
 LA English  
 CC 68-1 (Phase Equilibria, Chemical Equilibria, and Solutions)  
 Section cross-reference(s): 51  
 AB A method is presented for calculating the solubility of condensed, nonvolatile components in **supercrit. solvents** by treating the **supercrit. fluid**-phase mixture as an **expanded liquid**. The procedure is directly applicable to phase equilibrium calcns. associated with **extraction processes utilizing supercrit. solvents**. Two mixture parameters are required in the formulation for a binary system—an activity coefficient at infinite dilution for the heavy solute and a binary interaction parameter (i.e.  $k_{12}$  in the Redlich-Kwong equation of state). The advantage of this approach is that both mixture parameters exhibit consistent, predictable behavior for highly asym. mixts. in the vicinity of the critical region. The utility of this method is illustrated using exptl. data for the solubility of solid naphthalene in **supercrit. carbon dioxide** and in **supercrit. ethylene**.  
 ST hydrocarbon solv **supercrit solvent** calcn; heavy hydrocarbon solv **supercrit solvent**; carbon dioxide **supercrit** dissolv hydrocarbon  
 IT Solubility  
     (calcn. of, for heavy hydrocarbon solids in **supercrit. solvents**)  
 IT Hydrocarbons, properties  
     RL: PRP (Properties)  
         (solubility of solid, in **supercrit. solvents**)  
 IT 124-38-9, properties  
     RL: PRP (Properties)  
         (solubility in **supercrit.**, of heavy hydrocarbon solids)  
 IT 124-38-9, properties  
     RL: PRP (Properties)  
         (solubility in **supercrit.**, of heavy hydrocarbon solids)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

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L97 ANSWER 1 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:950310 HCPLUS  
 DN 140:6706  
 TI Electrostatic deposition of particles generated from rapid expansion of **supercritical** fluid solutions  
 IN Fulton, John L.; Deverman, George  
 PA Battelle Memorial Institute, USA  
 SO U.S. Pat. Appl. Publ., 12 pp.  
 CODEN: USXXCO  
 DT Patent  
 LA English  
 FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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PI US 2003222019	A1	20031204	US 2002-157626	20020528

PRAI US 2002-156970 20020528  
 AB A method is described for depositing a substance on a substrate, comprising forming a **supercrit.** solution of  $\geq 1$  **supercrit.** **solvent** and  $\geq 1$  **solute**, discharging the **supercrit.** solution through an orifice under conditions sufficient to form solid nanoparticles of the **solute** substantially free of the **supercrit.** **solvent**, and electrostatically depositing the nanoparticles onto the substrate. The nanoparticles may be charged to a first elec. potential and then deposited onto the substrate to form a film. The **solute** particles have a mean size of  $< 1 \mu\text{m}$ .  
 IT 124-38-9, **Carbon dioxide**, processes  
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses) (electrostatic deposition of particles from rapid **expansion** of **supercrit.** solns.)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

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L97 ANSWER 2 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:950308 HCPLUS  
 DN 140:6704  
 TI Electrostatic deposition of particles from rapid **expansion** of **supercritical** fluid solutions  
 IN Fulton, John L.; Deverman, George  
 PA Battelle Memorial Institute, USA  
 SO U.S. Pat. Appl. Publ., 13 pp.  
 CODEN: USXXCO  
 DT Patent  
 LA English  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2003222017	A1	20031204	US 2002-156970	20020528
PRAI	US 2002-156970		20020528		
AB	A method is described for depositing a substance on a substrate comprising forming a <b>supercrit.</b> solution of $\geq 1$ <b>supercrit.</b> <b>solvent</b> and $\geq 1$ <b>solute</b> , discharging the <b>supercrit.</b> solution through an orifice under conditions sufficient to form solid nanoparticles of the <b>solute</b> free of the <b>supercrit.</b> <b>solvent</b> , and electrostatically depositing the nanoparticles onto the substrate. The nanoparticles may be charged to a first elec. potential and then deposited onto the substrate to form a film. The <b>solute</b> particles have a mean size of $< 1 \mu\text{m}$ .				
IT	124-38-9, <b>Carbon dioxide</b> , processes 7440-37-1, Argon, processes 7440-63-3, Xenon, processes RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses) (electrostatic deposition of particles from rapid <b>expansion</b> of <b>supercrit.</b> solns.)				
RN	124-38-9 HCPLUS				
CN	Carbon dioxide (8CI, 9CI)	(CA INDEX NAME)			

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RN 7440-37-1 HCPLUS

CN Argon (8CI, 9CI) (CA INDEX NAME)

Ar

RN 7440-63-3 HCPLUS  
CN Xenon (8CI, 9CI) (CA INDEX NAME)

Xe

L97 ANSWER 3 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:915254 HCPLUS  
 DN 139:399673  
 TI Behavior of poly(methyl methacrylate)-based systems in **supercritical CO<sub>2</sub>** and **CO<sub>2</sub>** plus **cosolvent**: Solubility measurements and process assessment  
 AU Domingo, C.; Vega, A.; Fanovich, M. A.; Elvira, C.; Subra, P.  
 CS Instituto de Ciencia de Materiales de Barcelona, CSIC, Bellaterra, 08193, Spain  
 SO Journal of Applied Polymer Science (2003), 90(13), 3652-3659  
 CODEN: JAPNAB; ISSN: 0021-8995  
 PB John Wiley & Sons, Inc.  
 DT Journal  
 LA English  
 AB Microspheres based on synthetic polymers such as poly(Me methacrylate) (PMMA) and PMMA blends are known for their medical and optical applications. The development of methods for processing polymeric microspheres using a nontoxic **solvent**, like **supercrit. carbon dioxide** (SCCO<sub>2</sub>), is desirable. This work investigates the solubility and behavior of polymers (PMMA and PMMA/polycaprolactone blend) and **solutes** (cholesterol and albumin) in SCCO<sub>2</sub> and SCCO<sub>2</sub> + **cosolvent** (acetone, ethanol, and methylene chloride). The knowledge of solubility behavior of materials in SCCO<sub>2</sub> aids in the selection and/or design of the most appropriate technique for materials processing. Processing PMMA-based polymers with pure SCCO<sub>2</sub> leads to polymer swelling. The lack of polymer solubility in pure CO<sub>2</sub> precludes their micronization by the RESS (rapid expansion of **supercrit.** solns.) process, but on the other hand allows their impregnation. Polymer plasticization caused by CO<sub>2</sub> can be exploited in the PGSS (particles from gas-saturated solns.) process. Addition of a liquid **cosolvent** to CO<sub>2</sub> enhances the dissoln. of **solutes** and polymers. Precipitation of the studied polymers by **antisolvent** techniques seems feasible only by use of CO<sub>2</sub> + methylene chloride.  
 IT 124-38-9, Carbon dioxide, processes  
 RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)  
 (behavior of poly(Me methacrylate)-based systems in **supercrit. CO<sub>2</sub>** and **CO<sub>2</sub>** plus **cosolvent**)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

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## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
----------------------------	---------------	--------------	-------------	--------------------------	-----------------

Abraham, G	2000	282	44	J Macromol Mater Eng	HCAPLUS	
Alessi, P	1998			Proceedings of the 5		
Berens, A	1992	46	231	J Appl Polym Sci	HCAPLUS	
Burkoth, A	2000	21	2389	Biomaterials		
Castellani, S	1996			Ph D Thesis, Univers		
Chang, C	1997	131	243	Fluid Phase Equilib	HCAPLUS	
Condo, P	1994	325	23	J Polym Sci Part B:		
Cooper, A	2000	10	207	J Mater Chem	HCAPLUS	
Debenedetti, P	1993	24	27	J Controlled Release	HCAPLUS	
Domb, A	1994			Polymeric Site Speci		
Domingo, C	1997	10	39	J Supercrit Fluids	HCAPLUS	
Domingo, C	2001	21	147	J Supercrit Fluids	HCAPLUS	
Du, J	1997	43	223	J Controlled Release		
Engwicht, A	2000	21	1587	Biomaterials	HCAPLUS	
Ghaderi, R	1999	16	676	J Pharm Res	HCAPLUS	
Hubbell, D	1977	21	3035	J Appl Polym Sci	HCAPLUS	
Kim, H	1997	18	1175	Biomaterials	HCAPLUS	
Kosal, E	1992	5	169	J Supercrit Fluids	HCAPLUS	
Ksibi, H	1996	7	21	Adv Powder Technol	HCAPLUS	
Lee, S	1997	38	1317	Polymer	HCAPLUS	
Lin, W	2002	198	109	J Membr Sci	HCAPLUS	
Liu, G	1996	9	152	J Supercrit Fluids	HCAPLUS	
Lucien, F	2000	17	111	J Supercrit Fluids	HCAPLUS	
Magnan, C	1996		509	High Pressure Chem E	HCAPLUS	
McHugh, M	1994			Supercritical Fluid		
Middleton, J	2000	21	2335	Biomaterials	HCAPLUS	
Mishima, K	2000	46	857	AIChE J	HCAPLUS	
Reverchon, E	2000	18	239	J Supercrit Fluids		
Robinson, J	1987			Controlled Drug Deli		
Shieh, Y	1996	59	695	J Appl Polym Sci	HCAPLUS	
Shieh, Y	1996	59	707	J Appl Polym Sci	HCAPLUS	
Shine, A	1997			WO 9815348	HCAPLUS	
Siakumar, M	2000	46	29	React Funct Polym		
Siripurapu, S	2000	629	FF991	J Mater Res Soc Symp		
Subra, P	1997	131	269	Fluid Phase Equilib	HCAPLUS	
Subra, P	1998	12	261	J Supercrit Fluids	HCAPLUS	
Suzuki, K	1990	35	63	J Chem Eng Data	HCAPLUS	
Tams, J	1995	16	1049	J Biomaterials		
Thiering, R	2000	75	42	J Chem Technol Biote	HCAPLUS	
Vega-Gonzalez, A				J Chem Eng, to appear		
Vincent, M	1997	43	1838	AIChE J	HCAPLUS	
Walenkamp, G	1998			Biomaterials in Surg		
West, B	1998	69	911	J Appl Polym Sci	HCAPLUS	
Wissinger, R	1987	25	2497	J Polym Sci Part B:	HCAPLUS	
Wong, J	1986	2	29	Biotechnol Prog	HCAPLUS	
Yun, S	1991	30	2476	Ind Eng Chem Res	HCAPLUS	

L97 ANSWER 4 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:843045 HCAPLUS

DN 140:28251

TI Relationship between volume **expansion**, **solvent-power**,  
and **precipitation** in **GAS** processes

AU Striolo, Alberto; Elvassore, Nicola; Parton, Tiziana; Bertucco, Alberto

CS Dipt. di Principi e Impianti di Ingegneria Chimica, Universita di Padova,  
Padua, I-35131, Italy

SO AIChE Journal (2003), 49(10), 2671-2679

CODEN: AICEAC; ISSN: 0001-1541

PB American Institute of Chemical Engineers

DT Journal

LA English

AB Dilute solns. of Et cellulose (ETC) in acetone and of poly(ethylene oxide)  
(PEO) in Et acetate, acetonitrile, Et acetate-acetonitrile, and

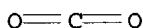
acetonitrile - water mixts. were expanded isothermally by compressed CO<sub>2</sub>. Onset precipitation pressures were visually measured through a windowed cell. Toward a rational understanding of the mol. mechanisms involved in gas antisolvent (GAS) processes, saturated-liquid-phase volume expansion and solvent power were monitored by UV-vis spectroscopy for the solvent mixts. considered in the precipitation expts. Ferrocene absorbance and phenol blue absorption-peak-wavelength shifts were used as probes to assess saturated-liquid-phase volume expansion and solvent power, resp. For the first time, a correlation between a microscopic bulk property, solvent power, and the onset precipitation pressure of a solute is reported. Because of preferential interactions with the dye (hydrogen bonds), the correlation breaks down when even small amts. of water are present in the solvent mixture. The results presented here suggest that UV-vis spectroscopy constitutes a valuable tool for understanding some phenomena related to supercrit.-fluid technol.

IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)  
(relationship between volume expansion, solvent  
-power, and precipitation in gas antisolvent  
processes)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



RETABLE

Referenced Author (RAU)	Year (R PY)	VOL (R VL)	PG (R PG)	Referenced Work (RWK)	Referenced File
Baggio, M	1998			Tesi di Laurea, Univ	
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bertucco, A	1999		231	Proc Int Meeting of	
Brennecke, J	2000			Proc Int Symp Superc	
Carlier, C	1993	39	876	AIChE J	HCAPLUS
Chang, C	1990	36	939	AIChE J	HCAPLUS
Day, C	1996	41	839	J Chem Eng Data	HCAPLUS
De la Fuente Badilla, J	2000	17	13	J Supercrit Fluids	HCAPLUS
Debenedetti, P	1987	42	2203	Chem Eng Sci	HCAPLUS
Debenedetti, P	1989	90	4528	J Chem Phys	HCAPLUS
Eberhardt, R	1997		1195	Liebigs Ann/Recueil	HCAPLUS
Eckert, C	1983	14	167	Fluid Phase Equilib	HCAPLUS
Eckert, C	1986	86	2738	J Phys Chem	
Elvassore, N	2002	42	223	J Chem Eng Data	
Elvassore, N	2001	90	1628	J Pharm Sci	HCAPLUS
Favari, F	2000	55	2379	Chem Eng Sci	HCAPLUS
Figueras, J	1971	93	3255	J Amer Chem Soc	HCAPLUS
Gallagher, P	1989	406		Amer Chem Soc Symp S	HCAPLUS
Kelley, S	1996	42	7	AIChE J	
Kim, S	1987	33	1603	AIChE J	HCAPLUS
Kim, S	1987	26	1206	Ind Eng Chem Res	HCAPLUS
Kolling, O	1973	45	160	Anal Chem	HCAPLUS
Kolling, O	1991	95	3950	J Phys Chem	HCAPLUS
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Morley, J	1999	103	11442	J Phys Chem A	HCAPLUS
Phillips, D	1993	32	943	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Sastri, V	1972	94	753	J Amer Chem Soc	HCAPLUS
Spilimbergo, S	2001	22	55	J Supercrit Fluids	
Subra, P	2000		921	Proc Meeting on Supe	

Subramaniam, B	1997	86	885	J Pharm Sci	HCAPLUS
Sun, Y	1992	114	1187	J Amer Chem Soc	HCAPLUS
Teja, A	2000	39	4442	Ind Eng Chem Res	HCAPLUS
Winters, M	1999	62	247	Biotechnol Bioeng	HCAPLUS
Yamaguchi, T	1993	109	9075	J Chem Phys	HCAPLUS

L97 ANSWER 5 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:295462 HCAPLUS

DN 138:306024

TI Vapor-Liquid Mass Transfer during **Gas Antisolvent Recrystallization**: Modeling and ExperimentsAU Lin, Cheng; Muhrer, Gerhard; Mazzotti, Marco; Subramaniam, Bala  
CS Institute of Process Engineering, ETH Swiss Federal Institute of Technology Zurich, Zurich, CH-8092, Switz.SO Industrial & Engineering Chemistry Research (2003), 42(10), 2171-2182  
CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

LA English

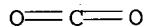
AB In batch **gas antisolvent (GAS) recrystn.**, the gradual addition of CO<sub>2</sub> to a liquid solution containing the solute causes the system pressure to rise and the volume of the liquid phase to **expand** substantially, eventually resulting in **solute precipitation**. The **expansion** rate depends on the rate of **antisolvent** addition and on the vapor-liquid mass-transfer rate and dets. the rate of supersatn. buildup in solution, which ultimately controls the particle formation process. The effect is studied of mass-transfer resistance on volume **expansion**, both theor. by development of a math. model of the mass-transfer phenomena under typical **GAS recrystn.** conditions and exptl. through volume **expansion** expts. (CO<sub>2</sub> in toluene) to assess the role of operating parameters such as stirring rate and aeration mode. A satisfactory agreement between model results and exptl. data is achieved in all cases.

IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)  
(modeling of vapor-liquid mass transfer in **gas antisolvent recrystn.**)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Albal, R	1983	27	61	Chem Eng J	HCAPLUS
Berends, E	1996	42	431	AIChE J	HCAPLUS
Berends, E	1994			Ph D Thesis, Technic	
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bird, R	1960			Transport phenomena,	
Bungert, B	1998	37	3208	Ind Eng Chem Res	HCAPLUS
de La Fuente, B	2000	17	13	J Supercrit Fluids	
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Irving, J	1977			National Engineering	
Jung, J	2001	20	179	J Supercrit Fluids	HCAPLUS
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCAPLUS
Kikic, I	1998	37	1577	Ind Eng Chem Res	HCAPLUS
Knaff, G	1987	21	151	Chem Eng Process	HCAPLUS
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS

Lucas, K	1981	53	959	Chem Ing Tech	HCAPLUS
Muhrer, G	2002	41	3566	Ind Eng Chem Res	HCAPLUS
Muhrer, G	2003			J Supercrit Fluids,	
Muhrer, G	2002			Ph D Thesis, ETH Zur	
Muller, M	2000	39	2260	Ind Eng Chem Res	
Nagata, S	1975			Mixing: Principles an	
Ng, H	1978	23	325	J Chem Eng Data	HCAPLUS
Peng, D	1976	15	59	Ind Eng Chem Fundam	HCAPLUS
Phillips, K	1973	51	371	Can J Chem Eng	HCAPLUS
Reid, R	1987			The properties of li	
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Schluter, V	1992	47	2357	Chem Eng Sci	HCAPLUS
Shariati, A	2002	23	195	J Supercrit Fluids	HCAPLUS
Shizimu, K	1998	191	178	J Cryst Growth	
Subramaniam, B	1997	88	885	J Pharm Sci	
Teramoto, M	1974	8	223	Chem Eng J	HCAPLUS
Werling, J	1999	16	167	J Supercrit Fluids	HCAPLUS
Werling, J	2000	18	11	J Supercrit Fluids	HCAPLUS
Wu, H	1995	50	2801	Chem Eng Sci	HCAPLUS

L97 ANSWER 6 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:269883 HCAPLUS

DN 139:87132

TI DELOS process: a **crystallization** technique using compressed fluids. 1. Comparison to the **GAS crystallization** method

AU Ventosa, N.; Sala, S.; Veciana, J.

CS Institut de Ciencia de Materials de Barcelona (CSIC), Campus Universitari de Bellaterra, Cerdanyola, 08193, Spain

SO Journal of Supercritical Fluids (2003), 26(1), 33-45  
CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB The depressurization of an **expanded** liquid organic solution (DELOS) **crystallization** technique is a new 1-step process, which uses a compressed fluid (CF) (e.g. CO<sub>2</sub>), for the straightforward production of sub-micron- or micron-sized **crystalline** particles. The driving force of a DELOS **crystallization** process is the fast, large and extremely homogeneous temperature decrease experienced by a solution, which contains aCF, when it is depressurized from a given working pressure to atmospheric pressure. In contrast to other already reported high-pressure **crystallization** techniques (RESS, **GAS**, PCA, PGSS), in a DELOS process the CF behaves as **co-solvent** over the initial organic solution of the **solute** to be **crystallized**. Through a DELOS process it is possible to produce fine powders of a compound provided that a system compound/organic **solvent**/CF' in a liquid 1-phase state is found. To compare DELOS and **gas anti-solvent** (**GAS**) procedures, 1,4-bis-(n-butylamino)-9,10-anthraquinone was **crystd** . from acetone/CO<sub>2</sub>' mixts. by both methods. The **crystn** . results obtained were analyzed upon the solubility behavior of 1,4-bis-(n-butylamino)-9,10-anthraquinone in acetone/CO<sub>2</sub>' mixts. with different composition. It will be seen how important is the knowledge of the **solute** solubility behavior in the CO<sub>2</sub>-**expanded** **solvent** to choose the most convenient **crystallization** technique (**GAS** like or DELOS) and the best operational parameters.Finally, it was exptl. determined which are the operational parameters that control the temperature decrease experienced in a DELOS **crystallization**. The results obtained were corroborated through thermodn. considerations.

IT 124-38-9, Carbon dioxide, processes

RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process); USES (Uses)

(comparison of depressurization (DELOS) process to **antisolvent (GAS)** crystallization methods of crystallization techniques using compressed fluids.)

RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

**RETABLE**

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Berends, E	1996	42	431	AIChE J	HCPPLUS
Bleich, J	1993	97	111	Int J Pharm	HCPPLUS
Bleich, J	1996	13	131	J Microencapsulation	HCPPLUS
Chang, C	1989	35	1876	AIChE J	HCPPLUS
Dixon, D	1993	50	1929	J Appl Polym Sci	HCPPLUS
Gallagher, P	1989	406	334	ACS Symposium Series	HCPPLUS
Gallagher, P	1992	5	130	J Supercritical Flui	HCPPLUS
Giacobbe, F	1992	72	277	Fluid Phase Equilibr	HCPPLUS
Jung, J	2001	20	179	J Supercrit Fluids	HCPPLUS
Kato, M	1991	24	767	Chem Eng Jap	HCPPLUS
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCPPLUS
Matson, D	1987	26	2298	Ind Eng Chem Res	HCPPLUS
Mawson, S	1997	13	1519	Langmuir	HCPPLUS
Mawson, S	1997	30	71	Macromolecules	HCPPLUS
Mohamed, R	1992	38	742	AIChE J	
Palakodaty, S	1998	1	275	Proceedings of the F	
Randolph, T	1993	9	429	Biotechnol Prog	HCPPLUS
Reverchon, E	1997		335	Proceedings of the F	
SYSTAT inc	1992			SYSTAT for windows,	
Shariati, A	2001		329	Proceedings of the S	
Tom, J	1991	7	403	Biotechnol Prog	HCPPLUS
Ventosa, N	2000			ES 01/00327	
Ventosa, N	2001	1	299	Crystal Growth and D	HCPPLUS
Weidner, E	1994	3	229	Proceedings of the T	
Yeo, S	1993	41	341	Biotechnol Bioeng	HCPPLUS

L97 ANSWER 7 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2003:182179 HCAPLUS

TI A green process to generate microparticles and nanoparticles

AU Sievers, Robert E.; Quinn, B. P.; Huang, Edward T. S.; Cape, S. P.; Alargov, D. K.; Villa, Joseph A.; Rinner, L.; Meresman, Helena V.; Mitchell, T. L., III; Vander Linden, B. J.

CS CIRES, Department of Chemistry and Biochemistry, and Center for Pharmaceutical Biochemistry, University of Colorado, Boulder, CO, 80309-0215, USA

SO Abstracts of Papers, 225th ACS National Meeting, New Orleans, LA, United States, March 23-27, 2003 (2003), ENVR-107 Publisher: American Chemical Society, Washington, D. C.

CODEN: 69DSA4

DT Conference; Meeting Abstract

LA English

AB Efficient methods for generating fine aerosols are very important for coating processes, thin film deposition, fine powder generation and pulmonary drug delivery. Traditionally, aerosols have been generated using liquid solvents containing environmentally objectionable organic compds. The process byproducts are toxic organic solvents and VOC gases. This paper describes a new green process for micronization and nanonization, Carbon Dioxide-Assisted Nebulization with a Bubble Dryer-, in which carbon dioxide is an

aerosolization agent and water is the **solvent** of choice. Aerosol is generated by intimately mixing dense **carbon dioxide** and an aqueous solution containing a **solute** of interest in a small volume tee at about 83 bar and room temperature. The resultant mixture is

**expanded** through a flow restrictor to form an aerosol, which is rapidly dried with **gaseous** nitrogen or air at 30 to 65°C to produce fine dry powders with diams. ranging from about 70 nm to 5 µm. Example of substances from which fine powders have been generated are anti-CD4 monoclonal antibody,  $\alpha$ 1-antitrypsin, doxycycline, amoxicillin, tobramycin sulfate, cromolyn sodium, albuterol sulfate, myo-inositol, ovalbumin, lactate dehydrogenase, trypsinogen, lysozyme, trehalose, sucrose, mannitol, potassium chloride and sodium chloride. The authors acknowledge the support of the Colorado Tobacco Research Program (Award No.1R-031) and NIH Leadership Training in Pharmaceutical Biotechnol. (HHS NIGMS Award Number 5 T32 GM08732-02).

- L97 ANSWER 8 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 2003:159447 HCAPLUS  
 DN 138:355605  
 TI Partial molar volume reduction of **solvent** for **solute** crystallization using **carbon dioxide** as **antisolvent**  
 AU Mukhopadhyay, Mamata  
 CS Department of Chemical Engineering, I.I.T. Bombay, Bombay, India  
 SO Journal of Supercritical Fluids (2003), 25(3), 213-223  
 CODEN: JSFLEH; ISSN: 0896-8446  
 PB Elsevier Science B.V.  
 DT Journal  
 LA English  
 AB The **gas antisolvent crystallization** (GASC) process using dense **carbon dioxide** (CO<sub>2</sub>) as **antisolvent** is particularly useful for purification and micronization of thermo-labile bioactive solid substances. Conventionally, the GASC process is characterized by the relative total volume **expansion** or the relative molar volume **expansion** of the solution. A new criterion is proposed in this work in terms of the relative partial molar volume reduction (RPMVR) of the **solvent** for selection of the **solvent** and the optimum process condition for the GASC process, as it directly gives a measure of the fraction of the dissolved **solute** **crystallized**. The **solute** solubility is proportional to the partial molar volume of the **solvent**, v<sub>2</sub> which drastically decreases at a high CO<sub>2</sub> dissoln. This is attributed to clustering of CO<sub>2</sub> mols. around the **solvent** mols. causing the loss of **solvent** power. This results in the desired **antisolvent** effect for lowering the **solute** solubility. v<sub>2</sub> has been calculated for a large number of **solvent**-CO<sub>2</sub> liquid mixts. using the Peng-Robinson equation of state. It has been observed that v<sub>2</sub> drastically reduces at a high value of x<sub>1</sub>, irresp. of the fact whether the **solvent** d. is higher or lower than that of the CO<sub>2</sub>. The **solute** solubility has been predicted from its value at the ambient pressure and the ratio of the partial molar volumes of the **solvent** with and without CO<sub>2</sub> dissolved in it. The predicted solubility of  $\beta$ -carotene in Et acetate with variation of x<sub>1</sub> at 298 K has been found to compare well with the exptl. observed trend of the GASC process.  
 IT 124-38-9, **Carbon dioxide**, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (partial molar volume reduction of **solvent** for **solute** crystallization by using **carbon dioxide** as **antisolvent**)  
 RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Badilla, J	2000	17	13	J Supercrit Fluids	
Chang, C	1990	36	939	AIChE J	HCAPLUS
Cocero, M	2000			Proceedings of the F	
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Mukhopadhyay, M	2000		50	Natural Extracts Usi	
Mukhopadhyay, M	2001			Proceedings of the 1	
Singh, S	2001			M Tech Dissertation,	

L97 ANSWER 9 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2002:453918 HCAPLUS

DN 137:146138

TI Measurements and modeling of the phase behavior of ternary systems of interest for the GAS process: I. The system carbon dioxide + 1-propanol + salicylic acid

AU Shariati, A.; Peters, C. J.

CS Faculty of Applied Sciences, Laboratory of Applied Thermodynamics and Phase Equilibria, Delft University of Technology, Delft, 2628 BL, Neth.

SO Journal of Supercritical Fluids (2002), 23(3), 195-208

CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB As a representative model system for the **gas-antisolvent** (GAS) process, the phase behavior of the ternary system **carbon dioxide** + 1-propanol + salicylic acid has been studied exptl. For this purpose, **carbon dioxide** has been chosen as the **anti-solvent gas**, 1-propanol as the organic **solvent**, and salicylic acid as the model drug. In each experiment, a solution of salicylic acid in 1-propanol was **expanded** using **carbon dioxide** as the **anti-solvent**. A synthetic method was used for measuring bubble point curves, and the solid (salicylic acid)-liquid boundaries. Three-phase equilibrium data solid (salicylic acid)-liquid-vapor were obtained from intersection of two-phase isopleths vapor-liquid and solid-liquid. Results are reported for this ternary system at **carbon dioxide** concns. ranging from 8.0 to 90.6 mol%, and within temperature and pressure ranges of 273-367 K and 1.0-12.5 MPa, resp. It has been observed that the **carbon dioxide** concentration significantly affects the optimum operational conditions of the GAS process, i.e. at lower concns. **carbon dioxide** acts as a **co-solvent**, while at higher concns. it acts as an **anti-solvent**. Also, it is shown that at a proper temperature, it is possible to **precipitate** most of the dissolved **solute** with only a small change of the pressure. The Peng-Robinson equation of state as modified by Stryjek and Vera (PRSV EOS) has been used to model the ternary system.

IT 124-38-9, **Carbon dioxide**, properties

RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)  
 (high-pressure phase equilibrium in **carbon dioxide**  
 /1-propanol/salicylic acid ternary mixts.)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

## RETABLE

Referenced Author (RAU)	Year (R PY)	VOL (R VL)	PG (R PG)	Referenced Work (RWK)	Referenced File
Chang, C	1990	36	939	AICHE J	HCAPLUS
Daubert, T	1989			Physical and Thermod	
De Fina, K	1999	44	1262	J Chem Eng Data	HCAPLUS
de la Fuente Badilla, J	2000	17	13	J Supercrit Fluids	HCAPLUS
Gauter, K	2000	171	127	Fluid Phase Equilib	HCAPLUS
Hanna, M	1997		325	Proceedings of the F	
Jaarmo, S	1997		263	Proceedings of the F	
King, M	1969			Phase Equilibrium in	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Liu, Z	2000	18	111	J Supercrit Fluids	HCAPLUS
Peng, D	1976	15	59	Ind Eng Chem Fund	HCAPLUS
Peters, C	1999	99	419	Chem Rev	HCAPLUS
Peters, C	1987	34	287	Fluid Phase Equilib	HCAPLUS
Peters, C	1993	85	301	Fluid Phase Equilib	HCAPLUS
Prausnitz, J	1986			Molecular Thermodyna	
Reverchon, E	2000	17	239	J Supercrit Fluids	HCAPLUS
Reverchon, E	1999	106	23	Powder Technol	HCAPLUS
Stephen, H	1963			Solubilities of Inor	
Stryjek, R	1986	64	820	Can J Chem Eng	HCAPLUS
Tavana, A	1991	284	5	AICHE Symposium Seri	
Thiering, R	2000	75	29	J Chem Technol Biote	HCAPLUS
Winters, M	1996	85	586	J Pharm Sci	HCAPLUS
Yeo, S	1993	41	341	Biotechnol Bioeng	HCAPLUS

L97 ANSWER 10 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:854939 HCAPLUS

DN 136:119925

TI Formation of Perfluoropolyether Coatings by the Rapid  
Expansion of Supercritical Solutions (RESS) Process.

Part 2: Numerical Modeling

AU Franklin, Randall K.; Edwards, Jack R.; Chernyak, Yury; Gould, Richard D.;  
Henon, Florence; Carbonell, Ruben G.CS Department of Mechanical and Aerospace Engineering and Department of  
Chemical Engineering, North Carolina State University, Raleigh, NC, 27695,  
USASO Industrial & Engineering Chemistry Research (2001), 40(26), 6127-6139  
CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

LA English

AB The rapid expansion of supercrit. solns. (RESS)

process is a promising method for the production of ultrafine powders and aerosols of narrow size distribution for coatings and other applications. In this article, part 2 of a two-part study, the nucleation and subsequent growth of 2500 Mw perfluoropolyether diamide (PFD) from supercrit. carbon dioxide (CO<sub>2</sub>) by expansion through a small-diameter nozzle is modeled in a three-stage, multidimensional fashion. The stages include a hydrodynamic solution, solvent-solute phase equilibrium analyses, and an aerosol transport model. The hydrodynamics model successfully captures the vapor-liquid transition that occurs as carbon dioxide is expanded to ambient conditions. Cloud-point pressures and equilibrium compns. of the separated solvent-solute system are determined and are used in a multidimensional aerosol transport model. This model incorporates various mechanisms influencing droplet growth.

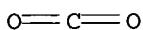
Parametric studies are conducted to investigate the influences of the interfacial tension, the equilibrium addition of carbon dioxide , and the diffusion coefficient on the predicted droplet diameter Turbulent coagulation in the ambient region downstream of the expansion nozzle is found to be the dominant mechanism responsible for the production of micron-sized droplets observed in companion expts.

IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
(formation of perfluoropolyether **coatings** by rapid expansion of **supercrit.** solns. (RESS) process. part 2: numerical modeling)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



**RETABLE**

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Chernyak, Y	2002	41	xxxxx	Ind Eng Chem Res	
Chernyak, Y	2000			Proceedings of the 5	
Debenedetti, P	1993	82	311	Fluid Phase Equilib	HCAPLUS
Edwards, J	2000	38	1624	AIAA J	HCAPLUS
Franklin, R	2000			Master's Thesis, Nor	
Friedlander, S	1977			Smoke, Dust, and Haz	
Hannay, J	1879	30	178	Proc R Soc London	
Harrison, K	1998	14	6855	Langmuir	HCAPLUS
Jung, J	2001	20	179	J Supercrit Fluids	HCAPLUS
Krukonis, V	1984			Presented at the AIC	
Ksibi, H	1996	10	69	Chem Biochem Eng Q	HCAPLUS
Kumar, S	1988	1	15	J Supercrit Fluids	HCAPLUS
Kwauk, X	1993	24	445	J Aerosol Sci	HCAPLUS
Lele, A	1992	38	742	AIChE J	HCAPLUS
Lindsay, J	1999			M S Thesis, North Ca	
Matson, D	1987	26	2298	Ind Eng Chem Res	HCAPLUS
Mawson, S	1995	28	3182	Macromolecules	HCAPLUS
McBride, B	1993			Coefficients for Cal	
Olchowny, G	1988	61	15	Phys Rev Lett	
Prausnitz, J	1986			Molecular Thermodyn	
Saffman, P	1956	1	16	J Fluid Mech	
Sanchez, I	1976	80	2352	J Phys Chem	HCAPLUS
Sanchez, I	1994		187	Models for Thermodyn	
Schaaf, P	1987	28	1930	Polymer	
Spalart, P	1992	1	5	Rech Aerosp	
Span, R	1996	25	1511	J Phys Chem Ref Data	
Wilcox, D	1998			Turbulence Modeling	
Wilke, C	1950	18	517	J Chem Phys	HCAPLUS
Zoller, P	1995		255	Standard Pressure-Vo	

L97 ANSWER 11 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:847154 HCAPLUS

DN 136:119892

TI Formation of Perfluoropolyether **Coatings** by the Rapid Expansion of **Supercritical** Solutions (RESS) Process.

Part 1: Experimental Results

AU Chernyak, Yury; Henon, Florence; Harris, Robert B.; Gould, Richard D.; Franklin, Randall K.; Edwards, Jack R.; DeSimone, Joseph M.; Carbonell, Ruben G.

CS Department of Chemical Engineering and Department of Mechanical and

Aerospace Engineering, North Carolina State University, Raleigh, NC, 27695, USA

SO Industrial & Engineering Chemistry Research (2001), 40(26), 6118-6126  
CODEN: IECRED; ISSN: 0888-5885

PB American Chemical Society

DT Journal

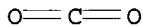
LA English

AB The rapid **expansion** of **supercrit.** solns. (RESS) process is a promising environmentally benign technol. for fine droplet or particle formation. The absence of organic **solvents** and narrow size distribution of RESS **ppts.** make this process attractive for polymer **coating** applications. This technique has been used to produce droplets of perfluoropolyethers from CO<sub>2</sub> solns. without the aid of **cosolvents** for the **coating** of porous materials applied in monumental and civil infrastructures. The present work is aimed at gaining an understanding of the relationship between droplet and spray characteristics and RESS process conditions. As such, a combined exptl./computational approach is applied to a representative binary system consisting of a low-mol.-weight perfluoropolyether diamide (PFD) dissolved in **supercrit.** CO<sub>2</sub>. Part 1 of this work presents phase equilibrium measurements and polymer droplet size characterizations under different operating conditions. The effects of temperature, **solute** concentration, and nozzle configuration on droplet and spray characterization and transfer efficiency are discussed. Part 2 of this work presents a multidimensional computational fluid dynamics model of the RESS **expansion** process and describes the use of the model in further analyzing and interpreting exptl. data.

IT 124-38-9, Carbon dioxide, uses  
RL: NUU (Other use, unclassified); USES (Uses)  
(**supercrit.**, **solvent**; formation of perfluoropolyether **coatings** by rapid **expansion** of **supercrit.** solns. process)

RN 124-38-9 HCPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Brennecke, J	1989	35	1409	AIChE J	HCAPLUS
Castelvetro, V	1998	11	551	Surf Coat Int	
Chang, C	1989	35	1876	AIChE J	HCAPLUS
Debenedetti, P	1990	36	1289	AIChE J	HCAPLUS
Debenedetti, P	1993	82	311	Fluid Phase Equilib	HCAPLUS
Desimone, J	1992	257	945	Science	HCAPLUS
Domingo, C	1997	10	35	J Supercrit Fluids	
Donohue, M	1995		152	Green Chemistry	
Franklin, R	2002	41	xxxxxx	Ind Eng Chem Res	
Henon, F	1999			Ph D Thesis, North C	
Kim, J	1996	12	650	Biotechnol Prog	HCAPLUS
Ksibi, H	1996	10	69	Chem Biochem Eng Q	HCAPLUS
Ksibi, H	1994		331	Proceedings of the 3	
Kwauk, X	1993	24	445	J Aerosol Sci	HCAPLUS
Lele, A	1994	33	1476	Ind Eng Chem Res	HCAPLUS
Lewis, J	1997		33	Met Finish	HCAPLUS
Liu, G	1997	30	293	J Chem Eng Jpn	HCAPLUS
Matson, D	1987	26	229	Ind Eng Chem Res	
Mawson, S	1995	28	3182	Macromolecules	
McHugh, M	1994			Supercritical Fluid	HCAPLUS

Mohamed, S	1989	35	325	AIChe J	
Muirhead, J	1974		248	Science and Technolo	
Piacenti, F	1994	68	227	J Fluorine Chem	HCAPLUS
Piacenti, F	1994	143	113	Sci Total Environ	HCAPLUS
Sanchez, I	1976	80	2352	J Phys Chem	HCAPLUS
Sanchez, I	1994		187	Models for Thermodyn	
Schaub, G	1995	8	318	J Supercrit Fluids	
Shim, J	1999	38	3655	Ind Eng Chem Res	HCAPLUS
Sianesi, D	1971	18	85	Wear	HCAPLUS
Span, R	1996	25	1511	J Phys Chem Ref Data	HCAPLUS
Tom, J	1991	22	555	J Aerosol Sci	HCAPLUS
Zoller, P	1995		255	Standard Pressure-Vo	

L97 ANSWER 12 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:792800 HCAPLUS

Correction of: 2000:812660

DN 135:322653

Correction of: 134:61420

TI Supercritical antisolvent micronization of some biopolymers

AU Reverchon, E.; Della Porta, G.; De Rosa, I.; Subra, P.; Letourneur, D.

CS Dipartimento di Ingegneria Chimica e Alimentare, Universita di Salerno, Fisciano, 84084, Italy

SO Journal of Supercritical Fluids (2000), 18(3), 239-245

CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

DT Journal

LA English

AB We proposed various biopolymers by semi-continuous **supercrit. antisolvent precipitation** (SAS) to evaluate the possibility of producing nano- and microparticles of controlled size and distribution. First, some liquid **expansion** curves were exptl. produced to study the general behavior of the ternary systems **antisolvent-solvent**-biopolymer. A condition that guarantees a successful SAS micronization is that **solute** does not modify the **expansion** curves of the **solvent-antisolvent** binary system. SAS expts. were performed by varying the process parameters; we mainly studied the influence of pressure, temperature and liquid solution concns. SEM images of the processed material were used to study morphologies, mean particle size and particle size distribution. We successfully processed by SAS dextran, poly(L-lactide) and poly(hydroxypropylmethacrylamide) by using DMSO and dichloromethane as liquid **solvents**.

## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Benedetti, L	1997	53	232	Biotech Bioeng	HCAPLUS
Benedetti, L	1995		221	Proceedings of the T	
Bertucco, A	1996		217	Proceedings of High	HCAPLUS
Bleich, J	1993	97	111	Internat J Pharm	HCAPLUS
Bleich, J	1994	106	77	Internat J Pharm	HCAPLUS
Bodmeier, R	1995	13	1211	Pharm Res	
Chou, Y	1997		55	Proceedings of the F	
Dillow, A	1997		247	Proceedings of the F	
Falk, R	1997		109e	Presented at AIChE A	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Mawson, S	1997	64	2105	J Appl Polym Sci	HCAPLUS
Randolph, T	1993	9	429	Biotech Progress	HCAPLUS
Rantakyla, M	1998	1	333	Proceedings of the 5	
Reverchon, E	1998	37	952	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1998	13	284	J Mater Res	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS

Reverchon, E	2000	17	239	J Supercrit Fluids	HCAPLUS
Reverchon, E	1999	102	129	Powder Technol	
Reverchon, E	1999		579	Proceedings of the F	
Saim, S	1996	13	S273	Pharm Res	
Thies, J	1998	45	67	Eur J Pharm Biopharm	HCAPLUS
Yeo, S	1993	26	6207	Macromolecules	HCAPLUS

L97 ANSWER 13 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:670920 HCAPLUS

DN 135:373704

TI Microparticle formation and crystallization rate of HMX with supercritical CO<sub>2</sub> antisolvent recrystallization

AU Cai, Jianguo; Zhou, Zhanyun; Deng, Xiu

CS Chemical Engineering Research Center, East China University of Science and Technology, Shanghai, 200237, Peop. Rep. China

SO Chinese Journal of Chemical Engineering (2001), 9(3), 258-261  
CODEN: CJCEEB; ISSN: 1004-9541

PB Chemical Industry Press

DT Journal

LA English

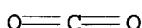
AB Microparticle formation and crystallization rate of 1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane (HMX) in acetone solution by using supercrit. carbon dioxide antisolvent (GAS) recrystn. were studied. Scanning electronic microscopy, X-ray diffraction and IR radiation were used to examine particle size, crystallinity and chemical structure. The β-HMX microparticle in different average size (2-9.5 μm) and with narrow size distribution were obtained by controlling the expansibility, expansion speed, initial concentration and temperature during recrystn. of HMX. The formation of nuclei is the a main cause of consumption of solute when the solution is expanded rapidly enough and the equilibrium concentration is lower, in which almost monodisperse microparticle can be obtained.

IT 124-38-9, Carbon dioxide, uses

RL: PEP (Physical, engineering or chemical process); PRP (Properties); TEM (Technical or engineered material use); PROC (Process); USES (Uses)  
(microparticle formation and crystallization rate of HMX with supercrit. CO<sub>2</sub> antisolvent recrystn  
. )

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Gallagher, P	1989		334	Supercritical Fluid	HCAPLUS
Gibbs, J	1957	1	322	Thermodynamics	
Hannay, J	1879	29	324	Proc Roy Soc	
Hoffsommer, J	1975	103	182	J Chromatography	HCAPLUS
Krukonis, V	1984			Ann Mtg AIChE	
Larsen, K	1986		73	Biotech Prog	
Nielsen, A	1964		350	Kinetic of Precipita	
Nyvlt, J	1971		189	Industrial Crystall	
Worthy, W	1981		16	C&E	
Yeo, S	1993	41	241	Biotech Bioeng	

L97 ANSWER 14 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 2001:195526 HCPLUS  
 DN 134:210028  
 TI Fine particle **coating** for release control by using RESS process  
 in fluidized bed  
 AU Wang, Tingjie; Tsutsumi, Atsushi; Jin, Yong  
 CS Dep. Chem. Eng., Tsinghua Univ., Beijing, 100084, Peop. Rep. China  
 SO Huagong Xuebao (Chinese Edition) (2001), 52(1), 50-55  
 CODEN: HUKHAI; ISSN: 0438-1157  
 PB Huaxue Gongye Chubanshe, Huagong Xuebao Bianjibu  
 DT Journal  
 LA Chinese  
 AB Fine particle **coating** was conducted by the rapid **expansion of supercrit.** fluid solution (RESS) in a fluidized bed for release control of some key component in core particles. The **supercrit.** carbon dioxide solution of paraffin was jetted into the fluidized bed of the core particles. The rapid phase change of the fluid solution from **supercrit.** state to **gas** results in a **solute** at high supersaturating state in the **solvent**, which forms a huge number of superfine nuclei in the jetting flow. The deposition of the superfine nuclei on the surface of the core particles leads to a thin layer **coating** of paraffin. The size of the superfine nuclei is in the order of 40 nm. A porous spherical particle was selected as the core particle, which carried a tracer component of a kind of dye. **Coating** level was examined by the tracer's release concentration in a **solvent** over a certain time. The state of **coating** was analyzed by measuring the average mass of coated particles and a SEM observation on the surface of coated particles. The rapid **expansion** of the **supercrit.** fluid solution causes a big temperature drop at the nozzle outlet. The low temperature of the nozzle outlet affects the phase of **carbon dioxide** and the properties of the superfine nuclei in the jetting flow, therefore it affects the particle **coating** process. The effect of temperature at nozzle inlet, an important parameter, on surface **coating** was investigated. Seal **coating** was formed on the core particle surface at higher temperature. Porous **coating** was formed on the core particle surface at lower temperature. The temperature of the nozzle inlet affects the nucleus size significantly. Higher temperature results in a bigger size of the superfine nuclei. By controlling the operation parameters, a satisfactory quality of **coated** particles was achieved.  
 IT 124-38-9, Carbon dioxide, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (fine particle **coating** for release control by using rapid **expansion of supercrit.** fluid soluble process in fluidized bed)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 15 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 2000:812660 HCPLUS  
 DN 134:61420  
 TI **Supercritical antisolvent** micronization of some biopolymers  
 AU Reverchon, E.; Della Porta, G.; De Rosa, I.; Subra, P.; Letourneur, D.  
 CS Dipartimento di Ingegneria Chimica e Alimentare, Universita di Salerno,  
 Fisciano, 84084, Italy  
 SO Journal of Supercritical Fluids (2000), 18(3), 239-245  
 CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.  
 DT Journal  
 LA English  
 AB We proposed various biopolymers by semi-continuous **supercrit.** **antisolvent precipitation** (SAS) to evaluate the possibility of producing nano- and microparticles of controlled size and distribution. First, some liquid **expansion** curves were exptl. produced to study the general behavior of the ternary systems **antisolvent-solvent**-biopolymer. A condition that guarantees a successful SAS micronization is that **solute** does not modify the **expansion** curves of the **solvent-antisolvent** binary system. SAS expts. were performed by varying the process parameters; we mainly studied the influence of pressure, temperature and liquid solution concns. SEM images of the processed material were used to study morphologies, mean particle size and particle size distribution. We successfully processed by SAS dextran, poly(L-lactide) and poly(hydroxypropylmethacrylamide) by using DMSO and dichloromethane as liquid **solvents**.

## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Benedetti, L	1997	53	232	Biotech Bioeng	HCAPLUS
Benedetti, L	1995		221	Proceedings of the T	HCAPLUS
Bertucco, A	1996		217	Proceedings of High	HCAPLUS
Bleich, J	1993	97	111	Internat J Pharm	HCAPLUS
Bleich, J	1994	106	77	Internat J Pharm	HCAPLUS
Bodmeier, R	1995	13	1211	Pharm Res	
Chou, Y	1997		55	Proceedings of the F	
Dillow, A	1997		247	Proceedings of the F	
Falk, R	1997		109e	Presented at AIChE A	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Mawson, S	1997	64	2105	J Appl Polym Sci	HCAPLUS
Randolph, T	1993	9	429	Biotech Progress	HCAPLUS
Rantakyla, M	1998	1	333	Proceedings of the 5	
Reverchon, E	1998	37	952	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1998	13	284	J Mater Res	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Reverchon, E	2000	17	239	J Supercrit Fluids	HCAPLUS
Reverchon, E	1999	102	129	Powder Technol	
Reverchon, E	1999		579	Proceedings of the F	
Saim, S	1996	13	S273	Pharm Res	
Thies, J	1998	45	67	Eur J Pharm Biopharm	HCAPLUS
Yeo, S	1993	26	6207	Macromolecules	HCAPLUS

L97 ANSWER 16 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN.

AN 2000:812654 HCAPLUS

DN 133:337029

TI Influence of thermodynamic behaviour and **solute** properties on homogeneous nucleation in **supercritical** solutions

AU Turk, Michael

CS Institut fur Technische Thermodynamik und Kaltetechnik, Universitat Karlsruhe (TH), Karlsruhe, D-76131, Germany

SO Journal of Supercritical Fluids (2000), 18(3), 169-184  
CODEN: JSFLEH; ISSN: 0896-8446

PB Elsevier Science B.V.

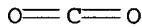
DT Journal

LA English

AB The knowledge about the thermodn. behavior of dilute **supercrit.** solns. is one of the basics for modeling processes, such as the formation of small particles by rapid **expansion of supercrit.** solns. (RESS). RESS allows the production of particles less than 1  $\mu\text{m}$  and RESS expts. show that particle size depends on **solvent**.

solute and preexpansion conditions. However, an understanding of the underlying phys. phenomena of the relationship between the process conditions and the mechanism of particle formation during RESS is still at an early stage. Because of that, there is a need to model the RESS process to get a better understanding of the influencing parameters. The calcns. show a steep increase at the beginning of the freejet reaching maximum theor. supersaturations of  $\approx 10^8$  and for an interfacial tension of 0.02 N m<sup>-1</sup> maximum nucleation rates of about  $10^{26}$  (cm<sup>-3</sup> s<sup>-1</sup>). In the present paper, the influence of the solubility of various **solutes** in **supercrit.** fluids and of the surface tension group ( $\sigma$  ·  $\sqrt{S}/3/k \cdot T$ ) of the diverse **solutes** on attainable nucleation rates under typical RESS operation conditions is investigated. The calcns. show that the nucleation rate is a sensitive function of the solubility and of the unknown surface tension group. Furthermore, it is shown that the classical nucleation theory is not able to describe the trend in particle size resulting from RESS expts. in a sufficient manner. Also, the present calcns. show that it is not possible to investigate homogeneous nucleation and coagulation sep. and that there is an enormous need for more reliable information about the **solute** properties.

IT 124-38-9, Carbon dioxide, processes  
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
 (thermodn. behavior and **solute** properties in homogeneous particle nucleation in **supercrit.** solns.)  
 RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Abraham, O	1981	75	402	J Chem Phys	HCPPLUS
Cihlar, S	1999	30	355	J Aerosol Sci	
Cihlar, S	1998		3	Proceedings of the W	
Debenedetti, P	1986	32	1253	Am Inst Chem Eng J	HCPPLUS
Debenedetti, P	1990	36	1289	Am Inst Chem Eng J	HCPPLUS
Foster, N	1991	30	1955	Ind Eng Chem Res	HCPPLUS
Helfgen, B	2000	110	22	J Powder Technol	HCPPLUS
Helfgen, B	1998		14	Proceedings of the A	
Jasper, J	1972	1	841	J Phys Chem Ref Data	HCPPLUS
Kodas, T	1986	111	102	J Colloid Interf Sci	HCPPLUS
Kruis, F	1993	19	514	Aerosol Sci Technol	HCPPLUS
Kwauk, X	1993	24	445	J Aerosol Sci	HCPPLUS
Lyman, W	1990			Handbook of Chemical	
Meyer, J	1998	3	31	Proceedings of the W	
Mohamed, R	1989	35	325	Am Inst Chem Eng J	HCPPLUS
Niekrawietz, M	1989			Dissertation, Univer	
Platzer, B	1989	10	223	Fluid Phase Equilib	
Pratsinis, S	1988	124	416	J Colloid Interf Sci	HCPPLUS
Preining, O	1998	29	481	J Aerosol Sci	HCPPLUS
Schmitt, W	1986	31	204	Chem Eng Data	HCPPLUS
Shaub, G	1995	8	318	J Supercrit Fluids	HCPPLUS
Singh, H	1993	32	2841	Ind Eng Chem Res	HCPPLUS
Springer, G	1978	14	281	Adv Heat Transfer	HCPPLUS
Tom, J	1991	22	555	J Aerosol Sci	HCPPLUS
Treffinger, P	1994	7	251	Fortschritt-Berichte	
Turk, M	1993			Dissertation, Univer	
Turk, M	1999	15	79	J Supercrit Fluids	HCPPLUS

Turk, M	1999	235	Proceedings of the I
Vargaftik, N	1996		Handbook of Physical

L97 ANSWER 17 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 2000:802758 HCAPLUS  
 DN 134:136546  
 TI Synthesis, Purification, and Micronization of Pharmaceuticals Using the Gas Antisolvent Technique  
 AU Warwick, B.; Dehghani, F.; Foster, N. R.; Biffin, J. R.; Regtop, H. L.  
 CS School of Chemical Engineering and Industrial Chemistry, University of New South Wales, Sydney, 2502, Australia  
 SO Industrial & Engineering Chemistry Research (2000), 39(12), 4571-4579  
 CODEN: IECRED; ISSN: 0888-5885  
 PB American Chemical Society  
 DT Journal  
 LA English  
 AB The synthesis, purification, and micronization of the nonsteroidal antiinflammatory Cu<sub>2</sub>(indomethacin)4L<sub>2</sub> (Cu-Indo); (L = DMF) has been investigated using DMF as the solvent and CO<sub>2</sub> as the antisolvent. The phase behavior of the binary system DMF + CO<sub>2</sub> and the ternary system DMF + CO<sub>2</sub> + Cu-Indo at 25, 30, and 40 °C and pressures up to 7.6 MPa was examined. The phase behavior of the ternary system DMF + CO<sub>2</sub> containing copper(II) acetate monohydrate (Cu-Acetate), indomethacin, or acetic acid and the quaternary system DMF + CO<sub>2</sub> containing Cu-Indo and either Cu-Acetate, indomethacin, or acetic acid at 25 °C and pressures up to 5.8 MPa was also examined to determine optimum synthesis conditions. The effect of variables such as reactant concentration, CO<sub>2</sub> wash volume, and rate of expansion on the purity and characteristics of the Cu-Indo produced in the synthesis was investigated. The recrystn. of Cu-Indo from DMF was investigated and the effect of the rate of expansion on the size of the particles produced was determined at 25 °C. It was found that Cu-Indo solubility in a DMF expanded solution decreased with increasing pressure and decreasing temperature. The solubility of Cu acetate in a DMF expanded solution was slightly increased as the pressure increased to 2.7 MPa and decreased rapidly at higher pressures. Upon addition of CO<sub>2</sub> to DMF + indomethacin saturated solns., a second liquid phase formed in the system and precipitation only occurred at pressures above 5.5 MPa. Acetic acid was found to remain soluble in the DMF expanded solution at the range of pressures and temps. examined. The addition of a second solute to the DMF + CO<sub>2</sub> + Cu-Indo solns. was found to significantly influence the phase behavior of the system. The solubility of Cu-Indo increased in the presence of acetic acid and Cu-Acetate and decreased in the presence of indomethacin. The product, Cu-Indo, with greater than 95% purity was produced in a single step at 25 °C. The presence of a slight excess of either reactant did not alter the purity of the Cu-Indo produced. The rate of expansion substantially varied the size and morphol. of the particles produced. Rapid expansion resulted in bipyramidal crystalline particles that were less than 10 μm in size. Slow expansion resulted in rhombic crystals with an average size of between 20 and 10 μm.  
 IT 124-38-9, Carbon dioxide, properties  
 RL: PEP (Physical, engineering or chemical process); PRP (Properties);  
 PROC (Process)  
 (preparation, purification, and micronization of pharmaceuticals using the gas antisolvent technique)  
 RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

## RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bungert, B	1997	69	298	Chem Ing Tech	HCAPLUS
Bungert, B	1998	37	3208	Ind Eng Chem Res	HCAPLUS
Chang, C	1991	7	275	Biotechnol Prog	HCAPLUS
Chang, C	1994	72	56	Can J Chem Eng	HCAPLUS
Chang, C	1995	40	850	J Chem Eng Data	HCAPLUS
Chang, C	1993	26	517	J Chem Eng Jpn	HCAPLUS
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Foster, N	1997		27	The 4th Internationa	
Gallagher, P	1989	406	334	ACS Symposium Series	HCAPLUS
Griffith, A	1999	38	411	Polym Plast Technol	HCAPLUS
Jianguo, C	1996	4	257	Chin J Chem Eng	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Liou, Y	1992	27	1277	Sep Sci Technol	HCAPLUS
Regtop, H	1990			WO 9014337	HCAPLUS
Regtop, H	1994			US 5310936	HCAPLUS
Regtop, H	1995			US 5466824	HCAPLUS
Reverchon, E	1999	15	1	J Supercrit Fluids	HCAPLUS
Savage, P	1995	41	1723	AIChE J	HCAPLUS
Shishikura, A	1994	42	1993	J Agric Food Chem	HCAPLUS
Shishikura, A	1997		51	The 4th Internationa	
Sorenson, R	1989			Progress in Medicina	
Subramaniam, B	1986	25	1	Ind Eng Chem Process	HCAPLUS
Tai, C	1998	44	989	AIChE J	HCAPLUS
Thiering, R	2000	75	29	J Chem Technol Biote	HCAPLUS
Weder, J	1999	38	1736	Inorg Chem	HCAPLUS

L97 ANSWER 18 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1999:698745 HCAPLUS

DN 132:266692

TI Gas antisolvent recrystallization of specialty chemicals

AU Muhrer, Gerhard; Mazzotti, Marco

CS Institut fur Verfahrenstechnik, ETH Zurich, Zurich, CH-8092, Switz.

SO International Symposium on Industrial Crystallization, 14th, Cambridge, United Kingdom, Sept. 12-16, 1999 (1999), 330-339 Publisher: Institution of Chemical Engineers, Rugby, UK.

CODEN: 68IRAJ

DT Conference; General Review; (computer optical disk)

LA English

AB A review with 84 refs. The need for the manufacturing of micron or sub-micron particles with narrow size distributions is gaining more and more importance in the production of specialty chems. and pharmaceuticals. In the last case microparticles are often intended for controlled drug release applications. There is therefore an increasing interest in developing technologies which, contrary to conventional techniques, allow microparticles with controlled particle size distribution and product quality to be produced under mild and inert conditions. **Supercrit** fluid technol., particularly when using **carbon dioxide**, offers promising possibilities for tackling this challenge, e.g., through the Rapid **Expansion of Supercrit**. Solns., **Precipitation** with Compressed **Antisolvent**, and **GAS** (**Gas Anti-Solvent**) techniques. In particular, **GAS recrystn.** exploits the low solubility of pharmaceutical compds. in **supercrit. carbon dioxide**, which

is used as **antisolvent** for the **solute** initially solubilized in a conventional **solvent**. Upon mixing by adding compressed **carbon dioxide** to the initial solution in a vessel, the solution is **expanded**, thus reducing its **solvent power**, and the **solute ppts.** Numerous exptl.

investigations have proved the attractiveness of these processes in terms of product quality; however, the understanding of their fundamentals and of the effects of individual process parameters is still very limited.

The development of applications of the **GAS recrystn.**

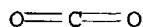
technol. requires that the gap between exptl. evidence and theor. understanding is filled.

IT 124-38-9, **Carbon dioxide**, uses

RL: NUU (Other use, unclassified); USES (Uses)  
(supercrit.; in **gas antisolvent**  
**recrystn.** of specialty chems.)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



#### RETABLE

Referenced Author (RAU)	Year (RPY)	VOL (RVL)	PG (RPG)	Referenced Work (RWK)	Referenced File
Alessi, P	1996	35	4718	Ind Eng Chem Res	HCAPLUS
Aniedobe, N	1997	30	2792	Macromol	HCAPLUS
Beckmann, W	1997	69	349	Chem Ing Tech	HCAPLUS
Benedetti, L	1997	53	232	Biotechnol Bioeng	HCAPLUS
Berends, E	1996	42	431	AIChE J	HCAPLUS
Bertucco, A	1998	44	2149	AIChE J	HCAPLUS
Bodmeier, R	1995	12	1211	Pharm Res	HCAPLUS
Bungert, B	1997	139	349	Fluid Phase Equilibr	HCAPLUS
Bungert, B	1998	37	3208	Ind Eng Chem Res	HCAPLUS
Catchpole, O	1996	12	309	Process Technology P	HCAPLUS
Chang, C	1989	35	1876	AIChE J	HCAPLUS
Chang, C	1990	36	939	AIChE J	HCAPLUS
Chang, C	1991	7	275	Biotechnol Progress	HCAPLUS
Chang, C	1994	72	56	Can J Chem Eng	HCAPLUS
Chang, C	1993	26	517	J Chem Eng Japan	HCAPLUS
Debenedetti, P	1990	36	1289	AIChE J	HCAPLUS
Debenedetti, P	1993	82	311	Fluid Phase Equilibr	HCAPLUS
Debenedetti, P	1993	24	27	J Controlled Rel	HCAPLUS
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Dixon, D	1993	39	127	AIChE J	HCAPLUS
Dixon, D	1993	50	1929	J Appl Polymer Sci	HCAPLUS
Domingo, C	1996	166	989	J Cryst Growth	HCAPLUS
Domingo, C	1997	10	39	J Supercrit Fluids	HCAPLUS
Falk, R	1997	44	77	J Controlled Rel	HCAPLUS
Falk, R	1998	15	1233	Pharm Res	HCAPLUS
Furuta, S	1995	148	197	J Cryst Growth	HCAPLUS
Gallagher, P	1989	406	334	ACS Symp Ser	HCAPLUS
Gallagher, P	1991	284	96	AIChE Symp Ser	
Gallagher, P	1992	5	130	J Supercrit Fluids	HCAPLUS
Griscik, G	1995	155	112	J Cryst Growth	HCAPLUS
Gromov, D	1998	108	4647	J Chem Phys	HCAPLUS
Gupta, P	1991	17	129	J Controlled Rel	
Jianguo, C	1996	4	257	Chin J Chem Eng	
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCAPLUS
Kim, J	1996	12	650	Biotechnol Progress	HCAPLUS
Kitamura, M	1997	178	378	J Cryst Growth	HCAPLUS
Knutson, B	1996	77	89	Drugs and the pharma	HCAPLUS

Kohn, J	1994	21	132	Proceed Intern Symp	
Kordikowski, A	1995	8	205	J Supercrit Fluids	HCAPLUS
Kwauk, X	1993	24	445	J Aerosol Sci	HCAPLUS
Larson, K	1986	2	73	Biotechnol Progress	HCAPLUS
Lele, A	1992	38	742	AIChE J	HCAPLUS
Lele, A	1994	33	1476	Ind Eng Chem Res	HCAPLUS
Liou, Y	1992	27	1277	Sep Sci Technol	HCAPLUS
Luna-Barcenas, G	1998	146	325	Fluid Phase Equilibr	HCAPLUS
Luna-Barcenas, G	1995	36	3173	Polymer	HCAPLUS
Matson, D	1986	1	242	Adv Ceram Mater	HCAPLUS
Matson, D	1987	21	109	Adv in Ceram	HCAPLUS
Mawson, S	1997	64	2105	J Appl Polymer Sci	HCAPLUS
Mawson, S	1997	13	1519	Langmuir	HCAPLUS
Mawson, S	1995	28	3182	Macromol	HCAPLUS
Mawson, S	1997	30	71	Macromol	HCAPLUS
Mawson, S	1997	38	2957	Polymer	HCAPLUS
Mishima, K	1998	61	179	Fukuoka University R	HCAPLUS
Mohamed, R	1989	406	355	ACS Symp Ser	HCAPLUS
Niehaus, M	1997	22	176	Prop Expl Pyrotech	HCAPLUS
Ohgaki, K	1990	3	103	J Supercrit Fluids	HCAPLUS
Reverchon, E	1998	37	952	Ind Eng Chem Res	HCAPLUS
Reverchon, E	1998	13	284	J Mater Res	HCAPLUS
Reverchon, E	1993	6	241	J Supercrit Fluids	HCAPLUS
Reverchon, E	1996	9	216	J Supercrit Fluids	HCAPLUS
Reverchon, E	1998	118	349	Stud Surf Sci Catal	HCAPLUS
Schmitt, W	1995	41	2476	AIChE J	HCAPLUS
Shaub, G	1995	8	318	J Supercrit Fluids	HCAPLUS
Shishikura, A	1994	42	1993	J Agric Food Chem	HCAPLUS
Shishikura, A	1992	5	303	Supercrit Fluids	HCAPLUS
Stejny, J	1998	39	4175	Polymer	
Subra, P	1996	12	217	Process Technology P	
Subramaniam, B	1997	86	885	J Pharm Sci	HCAPLUS
Tai, C	1998	44	989	AIChE J	HCAPLUS
Teipel, U	1997	22	165	Prop Expl Pyrotech	HCAPLUS
Thomasin, C	1998	87	259	J Pharm Sci	HCAPLUS
Thomasin, C	1998	87	269	J Pharm Sci	HCAPLUS
Tom, J	1992	514	238	ACS Symp Ser	
Tom, J	1991	7	403	Biotechnol Progress	HCAPLUS
Tom, J	1991	22	555	J Aerosol Sci	HCAPLUS
Tom, J	1992	7	9	J Supercrit Fluids	
Weidner, E	1996	12	217	Process Technology P	
Winters, M	1996	85	586	J Pharm Sci	HCAPLUS
Wubbolds, F	1997	667	242	ACS Symp Ser	HCAPLUS
Yeo, S	1993	41	341	Biotechnol Bioeng	HCAPLUS
Yeo, S	1994	83	1651	J Pharm Sci	HCAPLUS
Yeo, S	1993	26	6207	Macromol	HCAPLUS
Yeo, S	1995	28	1316	Macromol	HCAPLUS

L97 ANSWER 19 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1999:27755 HCAPLUS

DN 130:83612

TI Treatment of a substance with a dense fluid, especially with a supercritical fluid

IN King, Michael Blackshaw; Robertson, John

PA Smithkline Beecham PLC, UK; The University of Birmingham

SO PCT Int. Appl., 34 pp.

CODEN: PIXXD2

DT Patent

LA English

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 9858722	A1	19981230	WO 1998-GB1800	19980619

W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE,  
 DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG,  
 KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX,  
 NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT,  
 UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM  
 RW: GH, GM, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES,  
 FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI,  
 CM, GA, GN, ML, MR, NE, SN, TD, TG

AU 9881182 A1 19990104 AU 1998-81182 19980619  
 EP 991455 A1 20000412 EP 1998-930901 19980619

R: BE, CH, DE, ES, FR, GB, IT, LI, NL

JP 2002505617 T2 20020219 JP 1999-503993 19980619

PRAI GB 1997-12945 A 19970620  
 GB 1997-17344 A 19970816  
 WO 1998-GB1800 W 19980619

AB A process is disclosed for **precipitation** of a **solute** from a Dense Fluid Solvent. A solution of the **solute** in a Dense Fluid Solvent is **expanded** under conditions such that the Dense Fluid Solvent passes from the Dense Fluid Solvent region of its phase diagram into a 2-phase region of its phase diagram to cause **precipitation** of the **solute** from the solution. Apparatus for performing the process is also disclosed.

RETABLE

Referenced Author (RAU)	Year (R PY)	VOL (R VL)	PG (R PG)	Referenced Work (RWK)	Referenced File
British Nuclear Fuels P	1996			EP 0692289 A	HCAPLUS
Hewlett Packard Co	1990			EP 0384969 A	HCAPLUS
Jacques, L	1991			US 5011819 A	HCAPLUS
Moses, J	1988			US 4770780 A	HCAPLUS
Richard, S	1988			US 4734451 A	HCAPLUS

L97 ANSWER 20 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1998:686542 HCAPLUS

DN 129:262158

TI Fractional crystallization by gas antisolvent technique: theory and experiments

AU Bertucco, Alberto; Lora, Michele; Kikic, Ireneo

CS Istituto di Impianti Chimici, Universita di Padova, Padova PD, I-35131, Italy

SO AIChE Journal (1998), 44(10), 2149-2158

CODEN: AICEAC; ISSN: 0001-1541

PB American Institute of Chemical Engineers

DT Journal

LA English

AB The efficacy of CO<sub>2</sub> as an **antisolvent** was studied exptl. for the **precipitation** of naphthalene and phenanthrene from their solns. in toluene at 298 and 310 K. Phenanthrene was salted out of solution at every condition investigated, whereas naphthalene was never segregated as a solid phase. These behaviors are explained by a model representing the composition of the phases and supersatn. of the solution as functions of pressure. Based on results from ternary systems, expts. were performed with the quaternary system CO<sub>2</sub> -toluene-naphthalene-

phenanthrene: starting from an equimolar solution of the two solids in toluene, phenanthrene with a purity higher than 98.5% can be collected in the **precipitation** cell, while naphthalene with .apprx.13% of phenanthrene is recovered from the liquid phase after **expansion**.

The simulation of the process was able to account for the exptl. evidence.

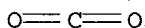
Although the **solutes** used do not have a practical application, a general method is outlined to exploit the possibility of using the **supercrit. antisolvent** technique for separation

IT 124-38-9, Carbon dioxide, uses

RL: NUU (Other use, unclassified); USES (Uses)

(fractional crystallization by gas antisolvent  
technique)

RN 124-38-9 HCAPLUS  
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



## RETABLE

Referenced Author (RAU)	Year (R PY)	VOL (R VL)	PG (R PG)	Referenced Work (RWK)	Referenced File
Catchpole, O	1996			Proc Int Symp on Hig	
Chang, C	1994	72	56	Can J Chem Eng	HCAPLUS
Dixon, D	1991	37	1441	AIChE J	HCAPLUS
Foster, N	1997			Proc 4th Int Symp on	
Gallagher, P	1989			Supercritical Fluid	
Hong, S	1992	74	133	Fluid Phase Equil	HCAPLUS
Kikic, I	1997	36	5507	Ind Eng Chem Res	HCAPLUS
Kikic, I	1997			Proc Int Symp on Sup	
Liang, M	1994			Proc Int Symp on Sup	
Liu, G	1996	35	4626	Ind Eng Chem Res	HCAPLUS
McHugh, M	1993			Supercritical Fluid	
Nagahama, K	1997			Proc Int Symp on Sup	
Shishikura, A	1994	42	1993	J Agric Food Chem	HCAPLUS
Shishikura, A	1992	5	303	J Supercrit Fluids	HCAPLUS
Shishikura, A	1991			Proc Int Symp on Sup	
Yeo, S	1993	41	341	Biotechnol and Bioen	HCAPLUS

L97 ANSWER 21 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1998:7381 HCAPLUS

DN 128:116698

TI Supercritical crystallization: designed  
crystallization? Rapid expansion of  
supercritical solutions (RESS) and gas  
antisolvent (GAS) and principal applications

AU Sanz Pastor, A. I.; Cocero, Alonso, M. J.

CS Dpto. Ingenieria Quimica, Universidad de Valladolid, Spain

SO Ingenieria Quimica (Madrid) (1997), 29(339), 183-190

CODEN: INQUDI; ISSN: 0210-2064

PB Ingenieria Quimica, S.A.

DT Journal; General Review

LA Spanish

AB The review, with 36 refs., covers methods of supercrit. fluid  
crystallization and discusses their possible uses in the pharmaceutical  
and polymer industries. Supercrit. crystallization methods  
can produce products with redefined particle sizes, narrow size  
distribution, absence of solvent occlusions, and residence times  
of seconds. In the RESS process (rapid expansion of  
supercrit. solns.), a solute dissolved in a  
supercrit. fluid ppts. to produce a sharp reduction in  
pressure and a following decline in solubility. The GAS (gas  
antisolvent) process uses a pressurized gas, under critical  
or quasi-critical (pressure and temperature close to the critical point)  
conditions,  
soluble in organic solvent and insol. in the solute, such  
that dissoln. provokes a volumetric expansion which reduces the  
solubility of the solute; the supercrit. fluid acts as an  
antisolvent, causing precipitation of solute.

## RETABLE

Referenced Author (RAU)	Year (R PY)	VOL (R VL)	PG (R PG)	Referenced Work (RWK)	Referenced File
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Berends, E	1994			PhD Thesis, Delft Un		
Brand, J	1988	166	139	Thin Solid Films	HCAPLUS	
Brunner, G	1990	30	191	International Chemic		
Chang, C	1989	35	1.876	AIChE Journal		
Chang, C	1991	7	275	Biotechnology Progre	HCAPLUS	
Cocero, M	1995	3	67	ALIMENTACION, TECNOL		
Cocero, M	1993		83	INGENIERIA QUIMICA		
Cocero, M	1995		169	INGENIERIA QUIMICA	HCAPLUS	
de La Osa, M	1991		251	INGENIERIA QUIMICA		
Debenedetti, P	1993	24	27	Journal of Controlle	HCAPLUS	
Desimone, J		265	356	Science	HCAPLUS	
Dixon, D	1993	39	127	AIChE Journal	HCAPLUS	
Eckert, C	1996	283	313	Nature		
Gallagher, P	1989		334	Supercritical Fluid	HCAPLUS	
Kordikowski, A	1995	8	205	The Journal of Super	HCAPLUS	
Krukonis, V	1989			Contract Rept		
Krukonis, V	1984			Paper 104f, AIChE me		
Larson, K	1986	2	73	Biotechnology Progre	HCAPLUS	
Lele, A	1992	38	742	AIChE Journal	HCAPLUS	
Lele, A	1994	33	1.476	Industrial Engineeri		
Lele, A	1990	31	677	Polym Prepr	HCAPLUS	
Loth, M	1986	32	265	International Journa		
Makita, T	1989		222	Proceedings of the I		
Matson, D	1986	1	242	Adv Cer Mat	HCAPLUS	
Matson, D	1987	21	109	Adv Ceram	HCAPLUS	
Matson, D	1989		480	Chemtech	HCAPLUS	
Medina, I	1993		443	Afinidad L		
Mohamed, R	1989	35	325	AIChE Journal	HCAPLUS	
Mohamed, R	1989		355	Supercritical Fluid	HCAPLUS	
Mueller, B	1989			DE 3744329	HCAPLUS	
Mullin, J	1993			"Crystallization", T		
Ohgaki, K	1990	3	103	The Journal of Super	HCAPLUS	
Schmitt, W	1995	41	2.476	AIChE Journal		
Tom, J	1991	7	403	Biotechnology Progre	HCAPLUS	
Tom, J	1993		239	Supercritical Engine		
Yeo, S	1993	41	341	Biotechnology and Bi	HCAPLUS	

L97 ANSWER 22 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1997:341799 HCAPLUS

DN 127:52671

TI Manufacture of microparticles by **crystallization** with highly compressed **gases**

AU Tschernjaew, Juri; Berger, Thomas; Weber, Andreas; Kummel, Rolf

CS Inst. Umwelt-, Sicherheits- Energietechnik e.V., Oberhausen, D-46047, Germany

SO Chemie-Ingenieur-Technik (1997), 69(5), 670-674  
CODEN: CITEAH; ISSN: 0009-286X

PB VCH

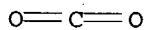
DT Journal

LA German

AB Two techniques for **precipitation** of **solutes** by addition of highly compressed **gases**, the **GAS** (**gas** **antisolvent** **crystallization**) and the **PCA** (particles with a compressed fluid **antisolvent**) process were studied using the **precipitation** of ascorbic acid or L-asparagine from saturated solns. in EtOH by addition of CO<sub>2</sub>. The **GAS** process gave particle sizes comparable to those of conventional **precipitation** and thermal **crystallization**, whereas the **PCA** process yielded particle sizes of the order of 1 μm and narrow size distribution. Disadvantages of the **GAS** process are the **crystallization** in the boundary layer combined with **precipitation** of polydisperse powders and the strong volume **expansion** of the liquid phase at the absorption of **gaseous**

**antisolvents** causing limited process capacity. The different mechanisms of precipitation depend on whether the **antisolvent** is a compressed, **supercrit.**, or liquefied **gas**.

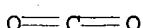
- L97 ANSWER 23 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1997:7897 HCAPLUS  
 DN 126:62342  
 TI Microparticle formation of HMX by **supercritical carbon dioxide antisolvent recrystallization**  
 AU Cai, Jianguo; Sun, Zhaohui; Ma, Hongxi; Liao, Xiaochun; Zhou, Zhanyun  
 CS Chem. Eng. Res. Center, ECU ST, Shanghai, 200237, Peop. Rep. China  
 SO Huadong Ligong Daxue Xuebao (1996), 22(5), 512-517  
 CODEN: HLIXEV  
 PB Huadong Ligong Daxue Xuebao Bianjibu  
 DT Journal  
 LA Chinese  
 AB The **recrystn.** ratio of 1, 3, 5, 7-tetranitro-1, 3, 5, 7-tetraazacyclooctane (HMX) in acetone, cyclohexanone, and dimethylsulfoxide solution using **supercrit. carbon dioxide antisolvent (GAS)** was compared. By using **GAS** process in acetone solution, microparticles of  $\beta$ -HMX within 2 .apprx. 13  $\mu\text{m}$  can be obtained. Effects of pressure, temperature, initial feed concentration of HMX **solute**, expansion speed of solution and growth of **crystal** on the **GAS** process have been studied. Under all exptl. pressures of 8.0 .apprx. 12.0 MPa tested, lower test temperature and lower concentration of feed solution were preferable for obtaining  $\beta$ -HMX and microparticles.  
 IT 124-38-9, **Carbon dioxide**, uses  
 RL: NUU (Other use, unclassified); TEM (Technical or engineered material use); USES (Uses)  
 (microparticle formation of HMX by **supercrit. carbon dioxide antisolvent recrystn.**)  
 RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



- L97 ANSWER 24 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1996:711759 HCAPLUS  
 DN 125:332821  
 TI Fine particle **coating** in a circulating fluidized bed by rapid **expansion of supercritical** fluid solutions  
 AU Tsutsumi, Atsushi; Nakata, Mitsutoshi; Mineo, Tomoko; Yoshida, Kunio  
 CS Dep. Chem. System Engineering, Univ. Tokyo, Tokyo, 113, Japan  
 SO Kagaku Kogaku Ronbunshu (1996), 22(6), 1379-1383  
 CODEN: KKRBAW; ISSN: 0386-216X  
 PB Kagaku Kogaku Kyokai  
 DT Journal  
 LA Japanese  
 AB Fine particle **coating** by rapid **expansion of supercrit. CO<sub>2</sub>** solns. of paraffins was performed in a circulating fluidized bed (50 mm i.d.) with an internal nozzle at the center of the riser. Microspheroidal catalyst particles (average particle size 56  $\mu\text{m}$ ) were used as the core particles. The **coating** mass and **coating** rates were measured by a sampling method. The effects of **gas** flow rate and **solute** concentration on **coating** rate and **coating** efficiency were examined  
 IT 124-38-9, **Carbon dioxide**, processes  
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical

process); PROC (Process); USES (Uses)  
 (supercrit., solvent; in coating of fine  
 particles in circulating fluidized beds by rapid expansion of  
 supercrit. solns.)

RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



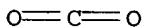
L97 ANSWER 25 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1996:616340 HCPLUS  
 DN 125:277764  
 TI Studying Activity Coefficients of Probe **Solutes** in Selected  
 Liquid Polymer **Coatings** Using Solid Phase Microextraction  
 AU Zhang, Zhouyao; Pawliszyn, Janusz  
 CS Department of Chemistry, University of Waterloo, Waterloo, ON, N2L 3G1,  
 Can.  
 SO Journal of Physical Chemistry (1996), 100(44), 17648-17654  
 CODEN: JPCHAX; ISSN: 0022-3654  
 PB American Chemical Society  
 DT Journal  
 LA English  
 AB The study of **solute-polymeric liquid solvent** interaction  
 contributes to the understanding of the fundamental principles of  
 chromatog. since liquid polymers are often used as stationary phases in  
**gas** chromatog. (GC) and high-performance liquid chromatog. (HPLC).  
 The knowledge of how a polymeric stationary phase interacts with different  
 types of compds. helps researchers to select and synthesize the right  
 phase for successful separation of mixts. in a time-efficient manner. The  
 development of a simple, cost effective, and time-efficient method for  
 studying **solute-solvent** interaction can aid  
 significantly the ever-expanding applications of chromatog. In  
 this work, a new approach, solid phase microextn. (SPME), is used for  
 investigations of activity coeffs. of the McReynolds probe **solutes**  
 in selected liquid polymers. The probe **solutes** are absorbed by an  
 immobilized liquid polymer phase **coated** on the outside surface of  
 a fused silica fiber, and quantitated by a GC technique using a com.  
 available GC column. The research in this study shows that activity  
 coeffs. measured by SPME are equivalent to those by the commonly used GC  
 method. This new method eliminates the need to prepare a GC column using  
 the polymer of interest as in the GC method and, thus, significantly  
 simplifies the whole measuring process. It also allows convenient  
 investigation of the prepared **coating** by other surface and  
 spectroscopic techniques.

L97 ANSWER 26 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1996:122671 HCPLUS  
 DN 124:274772  
 TI Crystallization of phenanthrene from toluene with **carbon**  
**dioxide** by the **GAS** process  
 AU Berends, Edwin M.; Bruinsma, Odolf S. L.; de Graauw, Jan; van Rosmalen,  
 Gerda M.  
 CS Lab. Process Equipment, Delft Univ. Technol., Delft, 2628 CA, Neth.  
 SO AIChE Journal (1996), 42(2), 431-9  
 CODEN: AICEAC; ISSN: 0001-1541  
 PB American Institute of Chemical Engineers  
 DT Journal  
 LA English  
 AB The **crystallization** of phenanthrene from toluene with CO<sub>2</sub> as  
 the **antisolvent gas** is described. In the **GAS**

process, a pressurized **gas** is dissolved into a liquid **solvent**, where it causes a volumetric **expansion** and lowers the solubility of the **solute**. Theor. models are presented for the liquid-phase **expansion** and the solubility as a function of pressure and temperature. The Nyvt theory for batch **crystallization** is adapted to predict the pressure profile in the **crystallizer** needed to maintain a constant supersatn. and growth rate. Generation of seeds is accomplished via a pressure pulse at the saturation pressure. The average particle

size of the phenanthrene could be varied from 160 to 540  $\mu\text{m}$ . Creation of seeds doubles the particle size and reduces the coefficient of variation significantly. The residual amount of toluene in the **crystals** without treatment is .apprx.70 ppm. The particles are agglomerates of phenanthrene **crystals**.

IT 124-38-9, **Carbon dioxide**, uses  
 RL: NUU (Other use, unclassified); USES (Uses)  
 (crystallization of phenanthrene from toluene by **gas**  
**antisolvent** process using)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



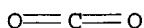
L97 ANSWER 27 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1996:4915 HCPLUS  
 DN 124:156296  
 TI An exact lattice model of complex solutions: chemical potentials depend on **solute** and **solvent** shape  
 AU Krukowski, Anton E.; Chan, Hue Sun; Dill, Ken A.  
 CS Dep. Pharmaceutical Chem., Univ. California San Francisco, San Francisco, CA, 94143-1204, USA  
 SO Journal of Chemical Physics (1995), 103(24), 10675-88  
 CODEN: JCPSA6; ISSN: 0021-9606  
 PB American Institute of Physics  
 DT Journal  
 LA English  
 AB For the theor. modeling of phys. transformations such as boiling, freezing, glassification, or mixing, it is necessary to know how the partition function of a system depends on its d. Many current treatments rely either on low d. **expansions** or they apply to very simple and sym. mol. shapes, like spheres or rods. Here the authors develop an exact anal. lattice theory of materials and mixts. that applies to arbitrarily complex mol. shapes over the full range of densities from **gas** to **crystal**. The approach is to compute partition functions for small nos. of shapes and to explore the dependence on d. by varying the volume of the system. Recently a question has been raised about whether entropies of dissoln. are better treated using classical solvation theories or Flory-Huggins theory. The authors explore this for a range of mol. sizes and shapes, from lattice squares and cubes, to rods, polymers, crosses, and other shapes. Beyond low densities, the entropic component of the chemical potential depends on shape due to the different degrees to which mols. "interfere" with each other. It was found that neither Flory-Huggins nor classical solvation theories is correct for all shapes. Mols. that are "articulated" are remarkably well treated by Flory-Huggins theory, over all densities, but globular mols. are qual. and quant. different, and are better treated by the classical chemical potential, consistent with expts. of Shinoda and Hildebrand. These results confirm that the Flory-Huggins theory differs from classical theory not because of mol. size differences per se; it accounts for the coupling between translations and conformational steric interference.

L97 ANSWER 28 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1995:26711 HCAPLUS  
 DN 122:107766  
 TI Solute deposition in a porous polymer matrix from rapid expansion of a supercritical solution  
 AU Bertucco, A.; Guarise, G. B.; Pallado, P.; Corain, B.  
 CS Istituto di Impianti Chimici, Universita di Padova, Padua, 35131, Italy  
 SO Chemical and Biochemical Engineering Quarterly (1994), 8(1), 11-16  
 CODEN: CBEQEZ; ISSN: 0352-9568  
 DT Journal  
 LA English  
 AB The rapid expansion of a supercrit. solution in a porous polymer matrix is carried out to obtain the deposition of the solute inside the structure. The sudden pressure reduction results in a strong supersatn., so that the formation of small solid particles can be achieved. The deposition of ferrocene crystallites on poly(N,N-dimethylacrylamide) is studied using CO<sub>2</sub> at temps. between 323-353 K and pressures from 18 to 22 MPa. A math. model is developed to represent the expansion of a real gas through the exit nozzle. Simulated and exptl. profiles for pressure and temperature are in agreement, so that the amount of precipitated solute and the phys. state of the solvent can be predicted.

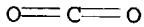
→ L97 ANSWER 29 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1994:587124 HCAPLUS  
 DN 121:187124  
 TI Precipitation of poly(L-lactic acid) and composite poly(L-lactic acid)-pyrene particles by rapid expansion of supercritical solutions  
 AU Tom, Jean W.; Debenedetti, Pablo G.; Jerome, Robert  
 CS Dep. Chem. Eng., Princeton Univ., Princeton, NJ, 08544, USA  
 SO Journal of Supercritical Fluids (1994), 7(1), 9-29  
 CODEN: JSFLEH; ISSN: 0896-8446  
 DT Journal  
 LA English  
 AB The rapid expansion of supercrit. solns. (RESS) was explored as a novel route to the formation of microparticles and microspheres useful in controlled drug delivery applications. Poly(L-lactic acid) was dissolved in supercrit. CO<sub>2</sub> with CHClF<sub>2</sub> as a cosolvent and precipitated by RESS. The polymers solubility and its mol. weight in solution were found to depend on processing time because of sample polydispersity. The morphol. of the precipitate (microparticles, microspheres, agglomerates, or dendrites) was examined as a function of the type of the expansion device (orifices or capillaries), pre-expansion temperature, and solvent composition. Dendrites were the most common morphol. when using orifices. Microspheres formation using capillaries occurred with low pre-expansion temps. and low length-to-diameter ratio. A one-dimensional fluid mech. model of the solvent's expansion in a capillary indicates that microspheres were formed preferentially when the fluid's exit d. was high, suggesting that substantial precipitation occurred outside the capillary. In the first comprehensive study of the effects of process conditions on the composite powders formed by RESS copptn., pyrene (a nonpolymeric fluorescent solute) was copptd. with poly(L-lactic acid) from supercrit. CO<sub>2</sub>-CHClF<sub>2</sub> solns. Fluorescence and transmission microscopy allowed the observation of pyrene in the coppt. These expts. showed clearly the uniform incorporation of pyrene microparticles within polymer microspheres, and thus, the feasibility of RESS as a technique for the copptn. of composite particles with multiple substances.  
 IT 124-38-9, Carbon dioxide, properties  
 RL: PRP (Properties)

(solvent; composite particles for controlled drug release  
copptn. by rapid expansion of **supercrit.** solns.)

RN 124-38-9 HCPLUS  
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



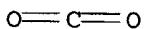
L97 ANSWER 30 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1994:194464 HCPLUS  
 DN 120:194464  
 TI Relative supersaturation ratio and separation factor in  
crystallization with high pressure CO<sub>2</sub>  
 AU Chang, Chiehming J.; Liou, Yuchung; Lan, Wen Jen  
 CS Dep. Chem. Eng., Natl. Chung-Hsing Univ., Taichung, 400, Taiwan  
 SO Canadian Journal of Chemical Engineering (1994), 72(1), 56-63  
 CODEN: CJCEA7; ISSN: 0008-4034  
 DT Journal  
 LA English  
 AB Crystallization in the presence of high-pressure gas as  
antisolvent could be applied for the recovery of valuable compds.  
from liquid solution A study of separation behavior is presented here for a  
mixture  
of anthracene and anthraquinone in cyclohexanone expanded with a  
gaseous antisolvent, CO<sub>2</sub>. The pressure range  
was 0.1-12 MPa; the temperature was either 292 or 313 K. Separation factors  
were  
obtained from the measured salted-out yields and the supersatn. of each  
solute could be also obtained for this pressure-tuning  
crystallization. The separation factor varied almost linearly with relative  
supersatn. ratio in the crystallization of anthracene-anthraquinone from  
cyclohexanone and CO<sub>2</sub>.  
 IT 124-38-9, Carbon dioxide, uses  
 RL: USES (Uses)  
 (in pressure-induced crystallization of anthracene and anthraquinone  
from cyclohexanone)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L97 ANSWER 31 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1994:94354 HCPLUS  
 DN 120:94354  
 TI Sample introduction in capillary **supercritical** fluid  
chromatography using sequential density gradient focusing and  
**solvent** venting  
 AU Liu, Zaiyou; Farnsworth, Paul B.; Lee, Milton L.  
 CS Dep. Chem., Brigham Young Univ., Provo, UT, 84602, USA  
 SO Journal of Microcolumn Separations (1991), 3(5), 435-42  
 CODEN: JMSEEJ; ISSN: 1040-7685  
 DT Journal  
 LA English  
 AB A technique was developed for large volume sample introduction in capillary  
**supercrit.** fluid chromatog. A 20-cm length of 200-μm i.d.  
capillary tubing was used as precolumn. The precolumn temperature could be  
easily controlled by passing an elec. current through an elec. conductive  
paint **coated** on its outer surface. During injection, the same

solvent was vented from the precolumn with CO<sub>2</sub> (gas) at 32 atm, while the precolumn was kept at room temperature. Solutes were transferred onto the head of the anal. column as a narrow band by d. gradient focusing, which was established with (a) a temperature gradient along the precolumn, (b) a rapid expansion of CO<sub>2</sub> from supercrit. fluid to gas, and (c) a temperature difference between the precolumn and the anal. column. This injection approach minimized solute mass discrimination and could be easily performed.

L97 ANSWER 32 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1994:33310 HCAPLUS  
 DN 120:33310  
 TI Purification of polycyclic aromatic compounds using salting-out separation in high-pressure carbon dioxide  
 AU Chang, Chiehming J.; Liou, Yuchung  
 CS Dep. Chem. Eng., Yuan Ze Inst. Technol., Taoyuan, 320, Taiwan  
 SO Journal of Chemical Engineering of Japan (1993), 26(5), 517-22  
 CODEN: JCEJAQ; ISSN: 0021-9592  
 DT Journal  
 LA English  
 AB Gas antisolvent crystallization has the potential for application in the recovery of valuable compds. from solution, and in the separation of solid-solid mixts. Exptl. data are presented for a mixture of anthracene and anthraquinone dissolved in cyclohexanone which was expanded by a gaseous antisolvent, CO<sub>2</sub>. The pressure range is 0.1-12 MPa, and the temperature 291-313 K. The relation of salted-out yield and normalized feed concentration gives an important parameter, the min. solubility, from which supersatn. can be defined for gas antisolvent crystallization. Effects of initial feed concns. of solid solutes, temperature, and pressure on the separation of anthracene and anthraquinone have also been studied.  
 IT 124-38-9, Carbon dioxide, uses  
 RL: USES (Uses)  
 (high-pressure, crystallization of polycyclic aromatic compds. using)  
 RN 124-38-9 HCAPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



O—C—O

L97 ANSWER 33 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN  
 AN 1993:452405 HCAPLUS  
 DN 119:52405  
 TI Manufacture of coated fine particles, especially, lanthanum oxide-coated silica particles  
 IN Kitagawa, Kazuo; Yamamoto, Seiichi; Moritoki, Masato  
 PA Kobe Steel Ltd, Japan  
 SO Jpn. Kokai Tokkyo Koho, 8 pp.  
 CODEN: JKXXAF  
 DT Patent  
 LA Japanese  
 FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 05057166	A2	19930309	JP 1991-246861	19910831
PRAI	JP 1991-246861		19910831		
AB	The process comprises dissoln. of a 1st solute (e.g., SiO <sub>2</sub> ) and 2nd solute (e.g., La <sub>2</sub> O <sub>3</sub> ) in 1st and 2nd solvents (e.g., both water) to form 1st and 2nd systems at supercrit. or				

subcrit. states, adiabatic **expansion** of the 1st system to form a 1st **solute** fine particles via supersatd. state, increasing the pressure to that of the 2nd system and mixing with the latter, then adiabatic **expansion** of the mixed system for **precipitating** and **coating** of the 2nd **solute** on the surfaces of the 1st **solute** fine particles via supersatd. state. The **coated** fine particles can be further **coated** with nth ( $n \geq 3$ ) **solutes** from nth solns. by the same operation.

L97 ANSWER 34 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1993:415024 HCPLUS  
 DN 119:15024  
 TI Three-phase separation process for solutions, especially seawater and waste liquids  
 IN Wilensky, Joseph  
 PA USA  
 SO U.S., 28 pp. Cont.-in-part of U.S. 5,084,187.  
 CODEN: USXXAM  
 DT Patent  
 LA English  
 FAN.CNT 2

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI US 5167838	A	19921201	US 1991-814564	19911230
US 5084187	A	19920128	US 1991-701452	19910515
PRAI US 1991-701452		19910515		

AB Seawater, brines, industrial wastewaters, and nonaq. industrial water liqs. are separated into potable water, concentrated brine, and purified solutes by dissolving a fluid, e.g., liquid or **gaseous** CO<sub>2</sub>, into the solution to produce a single-phase liquid, lowering the liquid temperature, and then performing a Joule-Thompson free **expansion** on the liquid. As a result, the liquid is separated into a evaporated gas phase mainly comprised of the **solute**, and a **crystallized** solid phase mainly comprised of the **solvent** (e.g., ice). Any remaining liquid is recycled. The ice can be melted and used in production of carbonated beverages. When the remaining liquid is a brine, MgCO<sub>3</sub> can be recovered from it.

IT 124-38-9, **Carbon dioxide**, occurrence  
 RL: OCCU (Occurrence)  
 (in seawater desalination and waste liqs. separation)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L97 ANSWER 35 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1993:220656 HCPLUS  
 DN 118:220656  
 TI Mysterious fine particles formed during the rapid **expansion** of **supercritical** water solutions  
 AU Tanaka, Yoshiyuki  
 CS Fac. Eng., Kobe Univ., Kobe, 657, Japan  
 SO Koatsuryoku no Kagaku to Gijutsu (1992), 1(4), 263-71  
 CODEN: KKGIE2; ISSN: 0917-639X  
 DT Journal  
 LA Japanese  
 AB Fine silica particles were produced by the rapid **expansion** of **supercrit.** water-SiO<sub>2</sub> solns. (RESS) at 723-823 K and pressures from 50 to 100 MPa. New spherical particles sprouting whiskers were also discovered in the autoclave after the RESS. The solubility of solids in

**supercrit.** fluids is a very sensitive function of temperature and pressure. Small changes of pressure result in large changes in d. and solvent power, because **supercrit.** fluids are highly compressible. The rapid expansion of **supercrit.** solns. can give rise to very large supersatn. ratios. Nucleation rates are determined by the competition among solvent expansion, cooling due to depressurization, and high supersatn. In order to control the product, morphol., the effects of exptl. parameters, such as preexpansion temperature and pressure, solute concentration, depressurization schemes, nozzle configuration, and sampling method on the product characteristics of materials, were investigated by means of SEM and x-ray diffraction anal. Control of particle size distribution is possible by regulating supersatn. ratio as well as suitable selection of preexpansion temperature and pressure. Unique features of the RESS process are discussed.

L97 ANSWER 36 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN

AN 1992:86953 HCPLUS

DN 116:86953

TI Manufacture of fine particles.

IN Moritoki, Masato; Kitagawa, Kazuo; Inoe, Yasuhiko

PA Kobe Steel, Ltd., Japan

SO Jpn. Kokai Tokkyo Koho, 9 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI JP 03271113	A2	19911203	JP 1990-67890	19900317
PRAI JP 1990-67890		19900317		

AB Fine particles are manufactured by dissoln. of a solute in a solvent of **supercrit.** or subcrit. state, adiabatic expansion in a closed high-pressure container, precipitation of the solute in the container, releasing of residual pressure from the container to atmospheric, then (or meanwhile) recovery of fine particles of the solute. Number and shape of the fine particles are controlled by controlling speed of the adiabatic expansion. Thus, SiO<sub>2</sub> fine particles was manufactured from aqueous solution of **supercrit.** state.

L97 ANSWER 37 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN

AN 1990:574645 HCPLUS

DN 113:174645

TI Homogeneous nucleation in **supercritical** fluids

AU Debenedetti, Pablo G.

CS Dep. Chem. Eng., Princeton Univ., Princeton, NJ, 08544, USA

SO AIChE Journal (1990), 36(9), 1289-98

CODEN: AICEAC; ISSN: 0001-1541

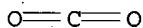
DT Journal

LA English

AB When a **supercrit.** solution is rapidly expanded, large solute supersatns. can be attained, and small particles are formed. The evolution of the homogeneous nucleation rate, work of nucleus formation, and critical nucleus size along different expansion paths is studied for the model system phenanthrene-CO<sub>2</sub>. Nucleation rates are the result of the competition among solvent expansion, cooling due to depressurization, and high supersatn. Although supersatns. can reach very high values (>10<sup>6</sup>), relatively flat nucleation rate profiles result due to cooling and expansion. For an interfacial tension of 0.02 N/m, computed nucleation rates never exceed 10<sup>4</sup>/s·cm<sup>3</sup>. A substantial fraction of the maximum nucleation rate is attained with partial decompression to >1 bar.

L97 ANSWER 38 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN

AN 1990:462140 HCPLUS  
 DN 113:62140  
 TI **Solvent expansion and solute solubility predictions in gas-expanded liquids**  
 AU Chang, Chiehming J.; Randolph, Alan D.  
 CS Dep. Chem. Eng., Univ. Arizona, Tucson, AZ, 85721, USA  
 SO AIChE Journal (1990), 36(6), 939-42  
 CODEN: AICEAC; ISSN: 0001-1541  
 DT Journal  
 LA English  
 AB The **expansion** of binary systems (e.g., PhMe-CO<sub>2</sub> and BuOH-CO<sub>2</sub>) in the miscible liquid-phase region and solubility of the **solute** (e.g., β-carotene in PhMe and acetaminophen in BuOH) in the liquid phase are studied. **Solvent expansion** at 298 K, solid solubility in the **gas antisolvent** addition for liquid-phase **precipitation** of solids, partial molar volume changes in the **gas antisolvent** addition process, and **crystallization kinetics** in the **gas antisolvent** addition **recrystn** are presented graphically and discussed.  
 IT 124-38-9, Carbon dioxide, properties  
 RL: PRP (Properties)  
 (expansion of, determination of, in **gas-expanded liqs.**)  
 RN 124-38-9 HCPLUS  
 CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L97 ANSWER 39 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1989:635821 HCPLUS  
 DN 111:235821  
 TI **Precipitation of microsize organic particles from supercritical fluids**  
 AU Chang, C. J.; Randolph, A. D.  
 CS Dep. Chem. Eng., Univ. Arizona, Tucson, AZ, 85721, USA  
 SO AIChE Journal (1989), 35(11), 1876-82  
 CODEN: AICEAC; ISSN: 0001-1541  
 DT Journal  
 LA English  
 AB The **precipitation** of organic particles from **supercrit.** fluids (SF) by **expansion** (SFX) has become an interesting alternative to milling without thermal decomposition. The rapid **expansion** produces a dramatic change of the **solute** supersatn. ratio that results in **precipitation** with a narrow particle-size distribution. It was found that β-carotene **ppts.** from SF ethylene and ethane have the feed material **crystallinity**. However, SF CO<sub>2</sub> reacted with β-carotene and did not give characteristic β-carotene x-ray spectra. The mean particle sizes of these **ppts.** were in the submicron range (.apprx.0.3 μm). Increased solubility was obtained by addition of PhMe as **cosolvent** in SF ethylene. The mean size of β-carotene particles remained unchanged if the PhMe concentration was <1.5 mol%. The SFX process appears to be in a single fluid phase when <1.5 mol% PhMe **cosolvent** is used.

L97 ANSWER 40 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1988:552362 HCPLUS  
 DN 109:152362  
 TI **Supercritical** fluid molecular spray thin films and fine powders  
 IN Smith, Richard D.

PA Battelle Memorial Institute, USA  
 SO U.S., 25 pp. Cont.-in-part of U.S. 4,582,731.  
 CODEN: USXXAM

DT Patent  
 LA English

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 4734451	A	19880329	US 1986-839079	19860312
	US 4582731	A	19860415	US 1983-528723	19830901
	CA 1327684	A1	19940315	CA 1988-556177	19880108
PRAI	US 1983-528723		19830901		
	US 1986-839079		19860312		

AB Solid films are deposited on surfaces or fine powders are formed by **supercrit.** fluid mol. spray in which a solution of the **supercrit.** fluid and the solid material as **solute** is formed, the solution is rapidly **expanded** through an orifice to produce a particulate spray and vaporized **solvent**, and the mol. spray is directed against a surface to deposit a film or it is discharged into a low pressure region to form a powder. The temperature of the **supercrit.** solution is selected and maintained for formation of the 2-phase system during **expansion** to control the porosity of the film or powder. Examples are discussed for the deposition of polystyrene films on Pt and fused silica, for the deposition of silica on Pt and glass, and for production of GeO<sub>2</sub> powders.

L97 ANSWER 41 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN

AN 1987:461383 HCPLUS

DN 107:61383

TI Production of powders and films by the rapid **expansion** of **supercritical** solutions

AU Matson, Dean W.; Petersen, Robert C.; Smith, Richard D.

CS Chem. Sci. Dep., Battelle, Pac. Northwest Lab., Richland, WA, USA

SO Journal of Materials Science (1987), 22(6), 1919-28

CODEN: JMTSAS; ISSN: 0022-2461

DT Journal

LA English

AB A process utilizing the rapid **expansion** of **supercrit.**

fluid solns. (RESS) is described for the manufacture of fine powders and thin films by the rapid nonequil. **precipitation** of nonvolatile compds. from dense **gas** solns. upon **expansion**. A variety of the fluid solution **expansion** parameters, including **solute** and **solvent** identity, **solute** concentration, **expansion** temperature, and **expansion** nozzle configuration, affect the product characteristics of materials formed during the RESS process. Conditions favoring thin film formation include very dilute solns. and short nozzles minimizing residence time during **expansion**. Particle formation is favored by more concentrated solns. The process produced products of widely varying morphol. by the adjustment of RESS parameters, and examples of SiO<sub>2</sub>, GeO<sub>2</sub>, and various polymeric materials are presented. Unique features of the RESS process relevant to other powder and film production methods are described and potential applications are discussed.

L97 ANSWER 42 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN

AN 1986:227104 HCPLUS

DN 104:227104

TI **Supercritical** fluid molecular spray film deposition and powder formation

IN Smith, Richard D.

PA Battelle Memorial Institute, USA

SO U.S., 15 pp.

CODEN: USXXAM

DT Patent

LA English

FAN.CNT 2

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 4582731	A	19860415	US 1983-528723	19830901
	JP 61500210	T2	19860206	JP 1984-503580	19840828
	JP 04019910	B4	19920331		
	CA 1260381	A1	19890926	CA 1984-461977	19840828
	US 4734451	A	19880329	US 1986-839079	19860312
PRAI	US 1983-528723		19830901		
	WO 1984-US1386		19840828		

AB Thin films are deposited, or fine powders are formed, by dissolving a solid material into a **supercrit.** fluid at an elevated pressure and then rapidly **expanding** the solution through a short orifice into a region of relatively low pressure. This produces a mol. spray which is directed against a substrate to deposit a solid thin film on it, or discharged into a collection chamber to collect a fine powder. Upon **expansion** and supersonic interaction with background **gases** in the low pressure region, the clusters of **solvent** are broken up and the **solvent** is vaporized and pumped away. **Solute** concentration in the solution is varied primarily by varying solution pressure. **Solvent** clustering and **solute** nucleation are controlled by manipulating the rate of **expansion** of the solution and the pressure of the lower pressure region.

L97 ANSWER 43 OF 44 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1980:189439 HCAPLUS

DN 92:189439

TI The effect of quasispherical and chainlike **solutes** on the nematic to isotropic phase transition in liquid **crystals**

AU Oweimreen, G. A.; Martire, D. E.

CS Dep. Chem., Georgetown Univ., Washington, DC, 20057, USA

SO Journal of Chemical Physics (1980), 72(4), 2500-10

CODEN: JCPSA6; ISSN: 0021-9606

DT Journal

LA English

AB The effects of **solute** mol. structure and **solvent** mol. structure on nematic phase stability in dilute binary mixts. of nonmesomorphic **solutes** and nematogenic **solvents** were studied. Addition of the perturbing **solute** to the liquid-crystal **solvent** leads to depression of the nematic-isotropic (NI) transition temperature and formation of a two-phase region. Directly determined moduli of the slopes,  $\beta_n$  vs. **solute** mol fraction ( $x_2$ ) diagrams are reported for quasispherical and chainlike **solutes** with two nematogenic **solvents**. The systems studied were the quasispheres Et4C (tetraethylmethane) and R4Sn ( $R = CH_3, C_2H_5, C_3H_7$  and  $C_4H_9$ ) and the chains ( $n-C_8H_{18}$  through  $n-C_{14}H_{30}$ , mixed with MBBA and p-n-pentyl-p'-cyanobiphenyl (5CB)). Also reported are indirectly determined  $\beta_{n\infty}$  and  $\beta_{i\infty}$  values (limit as  $x_2 \rightarrow 0$ ), using a novel approach combining differential scanning calorimetry (for the pure **solvent** contribution) and gas-liquid chromatog. (for the solution contribution), for Et4C and  $n-C_5H_{12}$  through  $n-C_{11}H_{24}$ , with MBBA, 5CB, p-azoxyanisole, and p,p'-di-hexyloxyazoxybenzene. For the systems in common, the average difference between the directly and indirectly determined  $\beta$  values is .apprx.10%, the comparison suggests slight curvature of the phase boundary lines. The exptl.  $\beta$  values, as a function of increasing **solute** size, double (roughly) for the quasispheres and increase only slightly for the chains, reflecting the concurrent behavior of the solution contribution to  $\beta$ . The thermodn. results for the quasispherical **solutes** are compared with predicted values from statistical-mech. theories based on rigid-rod **solvent** mols.: (1) lattice model, (2) virial **expansion** treatment, (3) mol.-field model (after Maier and Saupe), and (4) van der

Waals model. All four models correctly predict the observed trend of increasing  $\beta_n$  and  $\beta_i$  with increasing **solute** size and yield predicted slopes which are within a factor of 2 of experiment. All are deficient to a minor or major extent in their predictions of the **solvent** and solution contributions to the  $\beta$  values. The more tractable lattice model is used to examine the chainlike **solutes** and the effect of **solvent** end-chain flexibility. It correctly predicts the qual. features of the observed dependence of  $\beta$  on **solute** size for the different **solute** structures (including rigid-rod **solutes**) and indicates that dissolved n-alkane **solutes** have appreciable (effective) chain flexibility in nematic **solvents**. Incorporation of some **solvent** end-chain flexibility in the lattice model markedly improves agreement with experiment, primarily through better quant. prediction of the solution contribution.

L97 ANSWER 44 OF 44 HCPLUS COPYRIGHT 2004 ACS on STN  
 AN 1920:7933 HCPLUS  
 DN 14:7933  
 OREF 14:1468d-i,1469a-c  
 TI Some new hypotheses as to different states of matter  
 AU Bacon, N. T.  
 CS Peace Dale, RI  
 SO Journal of Physical Chemistry (1919), 23, 469-77  
 CODEN: JPCHAX; ISSN: 0022-3654  
 DT Journal  
 LA Unavailable  
 AB B. is not "satisfied with the current reason given to explain why mols. condense from a vessel filled with the saturated vapor when the temperature is reduced, for this same reduction should cause a reduction of pressure, even without condensation." After a consideration of the properties of a liquid and its vapor as they approach and pass the critical point, he asks, "is it not a fair inference, in view of these things, that in the condition of a true **gas** the spheres of influence of mols. decrease with advancing temperature so as to allow a free path and thus cause them to follow Boyle's law?". If in the **gaseous** condition the diameter of the sphere of influence of the mol. is thus an inverse function of the temperature, "we should find a probability that in the vapor condition at temps. below the critical the mol. would continue to **expand**. If this is true we should have a direct explanation of the separation of condensate whenever a saturated vapor is cooled under constant volume. There would no longer be room in their **gaseous** state for all the **expanding** mols. so that some of them would be obliged to go into the less bulky liquid form." A further development of this conception has grown out of the consideration of the very small solubility of BaSO<sub>4</sub>. The question is raised, "how (according to Calvert's determination) one single ion of Ba, in the presence of a corresponding ion of SO<sub>4</sub>, can affect simultaneously 10,000,000 mols. of H<sub>2</sub>O as to deprive every one of them of the power to dissolve any more BaSO<sub>4</sub>?" Regarding this problem he says, "I find the easiest explanation in assuming a virtual **expansion** of the mols. of the **solute** so as practically to occupy all the inter-mol. space of the **solvent** in much the same way in which I have supposed mol. in the volatile conditions to increase the diameter of the spheres of influence of their mols. as the temperature falls." Later he states, "that by way of explanation I find myself reduced to the conception of the BaSO<sub>4</sub> breaking up into an enormous number of electrons, or emanations of which electrons are built, each having the characteristic periodicity of BaSO<sub>4</sub> (and not solely of any constituent thereof) and that these so permeate the **solvent** that each mol. of this is in some way in

contact, periodic at least, with such particles, so as to maintain an equilibrium relation." "Colloidal solns. are merely individual mols. held in suspension and carry a current only mechanically through a menstruum which does not dissolve them. They take a charge by metallic conduction and thus are repelled from one pole and attracted to the other." "Hydrolysis represents a condition where the complicated periodicity of the salt becomes too extended, so that part of the **solute** loses coherence and the fractions revert to their simpler (though related) periodicity, each in its own condition, as if the other were not present. These conditions are quite different from those of electrolysis. In this the ions exist as atoms combined with charges of electricity (instead of complementary atoms) to make virtual mols. suspended in the menstruum much as are the metallic mols. in colloidal solns. and very different from the clouds of diffused electrons or emanations filling intermol. spaces which by my theory make a continuity of particles of the **solute** roughly answering to that of Bragg for matter in **crystalline** form. This involves recognizing inherent differences between **solvent** and **solute**. In many cases substances are mutually soluble, so that each acts both (or either) as **solvent** and (or) **solute**; in other cases one has a distinctly different type of action from the other." Regarding the more rapid **expansion** of a liquid as it approaches the critical point, there is suggested "the possibility that the rapid increase in volume may be due to mols. in the vapor state dissolved as vapor by other mols. of the same kind in the liquid state. This is analogous to Richards' explanation of the action of water between 0° and 4° as due to solution of ice mols. as such and, like  $S\mu$  in  $S\lambda$ ."

=> => fil wpix  
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FILE LAST UPDATED: 5 MAR 2004 <20040305/UP>  
MOST RECENT DERWENT UPDATE: 200416 <200416/DW>  
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THE TIME RANGE CODE WILL ALSO CHANGE FROM 018 TO 2004.  
SDIS USING THE TIME RANGE CODE WILL NEED TO BE UPDATED.  
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=> d all abeq tech abex tot

L116 ANSWER 1 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN  
AN 2004-068695 [07] WPIX  
CR 2002-099223 [14]; 2002-547217 [58]; 2002-547218 [58]; 2002-664436 [71];  
2003-267551 [26]; 2003-447308 [42]  
DNN N2004-055254 DNC C2004-028225

TI Collecting samples comprises controlling e.g. pressure of stream to improve separation of monophasic fluid into **gaseous** and liquid phases, **expanding** stream by directing through **expansion** space, and retaining liquid in collection device.

DC B04 J04 S03

IN BENTE, P F; BERGER, T A; FOGELMAN, K D; KLEIN, K; NICKERSON, M; STAATS, L T

PA (BERG-N) BERGER INSTR INC; (BENT-I) BENTE P F; (BERG-I) BERGER T A; (FOGE-I) FOGELMAN K D; (KLEI-I) KLEIN K; (NICK-I) NICKERSON M; (STAAT-I) STAATS L T

CYC 32

PI US 2002139752 A1 20021003 (200407)\* 29p B01D011-00 <--  
EP 1348956 A2 20031001 (200407) EN G01N030-06  
R: AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LI LT LU LV  
MC MK NL PT RO SE SI SK TR  
US 6632353 B2 20031014 (200407) B01D015-08 <--

ADT US 2002139752 A1 CIP of US 2000-607316 20000626, US 2002-113599 20020329;  
EP 1348956 A2 EP 2003-4475 20030227; US 6632353 B2 CIP of US 2000-607316  
20000626, US 2002-113599 20020329

FDT US 2002139752 A1 CIP of US 6413428; US 6632353 B2 CIP of US 6413428

PRAI US 2002-113599 20020329; US 2000-607316 20000626

IC ICM **B01D011-00**; **B01D015-08**; G01N030-06  
ICS G01N030-16; G01N030-28; G01N030-80

AB US2002139752 A UPAB: 20040128  
NOVELTY - Collecting samples from flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid comprising controlling the pressure, temperature and velocity of the flow stream to improve separation of a monophasic fluid mixture into separate **gaseous** and liquid phases and **expanding** by directing through an **expansion** space, is new.

DETAILED DESCRIPTION - Collecting samples from a flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid comprising controlling the pressure, temperature and velocity of the flow stream to improve separation of a monophasic fluid mixture into separate **gaseous** and liquid phases, **expanding** the flow stream by directing it through an **expansion** space, and retaining the liquid phase in a collection device, is new.

INDEPENDENT CLAIMS are also included for:

(1) collecting samples comprising injecting the samples into a flow stream, controlling the pressure, temperature and velocity of the flow stream to improve separation of a monophasic fluid mixture into separate **gaseous** and liquid phases, **expanding** the flow stream by directing it through an **expansion** space in a flow line carrying the flow stream, and retaining the liquid phase in a collection device;

(2) a system for collecting samples from a flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid, comprising a flow line creating a space in which the flow stream moving through the line is **expanded** and the linear velocity of the flow stream is slowed, and a collection device downstream of the space in the flow line, in which the liquid phase from the flow line is retained; and

(3) a further system for collecting samples from a flow stream containing a mixture of highly compressed **gas**, compressible liquid or supercritical fluid and a relatively incompressible liquid, comprising an injection valve for injecting discrete samples into the flow stream, a separation device to elute **solutes** of the samples, a detector to detect the concentrations of the **solutes** in the flow stream, a phase separation stage to control the pressure, temperature and velocity of the flow stream to improve separation, comprising a series of heaters and transfer lines to separate a monophasic flow stream into liquid and **gas** phases, an **expansion** space in the flow

stream sized to create a point of **expansion** of the flow stream and in which the linear velocity of the flow stream is slowed, and at least one collection device to retain the liquid phase.

**USE** - The method is used for chromatography, e.g. preparative and analytical supercritical fluid chromatography (SFC) or supercritical fluid extraction for a liquid phase SFC collection system.

**ADVANTAGE** - The process efficiently separates liquid and **gas** phases in a flow stream upstream of a collection vessel without additional pressure schemes or **solvent** extraction imposed on the flow stream. Samples are repeatedly injected into the mobile phase flow stream and collected into large-volume containers, allowing longer unattended run times and cost-efficient sample purification and recovery.

**DESCRIPTION OF DRAWING(S)** - The figure shows a schematic flow diagram of a supercritical fluid chromatography system and collection system including a sample cassette.

- Thermally regulated transfer tube 12
- Back-pressure regulator 14
- Heaters 16, 18, 20
- Valve system 22
- Waste stream container 26
- Transfer tubing lines 28
- Cassette lid 30
- Discrete chambers 32
- Waste transfer line 34
- Test tube vial 36
- Liquid phase 38
- Discharge lines 40
- Pressure relief switch 42
- Molded frame 44, 46
- Butterfly latches 56
- Restrictive transfer line 72

Dwg.1/15

FS	CPI EPI
FA	AB; GI
MC	CPI: B11-C06; B11-C09; J04-B01C EPI: S03-E09C; S03-E13B2
TECH	UPTX: 20040128

**TECHNOLOGY FOCUS - INSTRUMENTATION AND TESTING - Preferred Process:**

**Expanding** the flow stream comprises directing the stream through large bore tubing with an internal diameter sufficient to slow the linear speed of the flow stream, or comprises directing the flow stream through a chamber with an internal space sufficient to slow the linear speed of the flow stream.

Retaining the liquid phase in a collection device includes using a discrete collection container to receive the liquid phase, in which the container has an exit port for discharging waste products. The flow stream discharges from a flow line inside the collection device at an angle less than horizontal and at a tangential angle to the inner wall of the container. The process further comprises detecting the volume of the liquid phase in the collection device and stopping the flow stream filling the device when the liquid phase reaches a threshold level. The process is performed under approximate isocratic conditions.

The process further comprises injecting the samples into the process at a frequency such that a second sample injection begins elution of a sample **solute** after a first sample injection completes elution of the same **solute** within the first injection, but prior to the first sample completing an entire chromatographic process.

Determining the frequency of the sample injections comprises detecting the eluted **solutes** in the flow stream, determining time periods for elution of the **solutes** from the time of injection to the beginning of elution, determining the time periods from the start to finish of an eluted **solute** concentration peak, and automatically collecting the liquid phase containing the eluted **solutes** from

the repetitive sample injections into the collection device based on the time periods.

The process further comprises retaining the liquid phase in the collection device according to a start/stop signal from a detection device that is sent through a system controller for each of the sequential series of injections.

L116 ANSWER 2 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STM  
 AN 2003-531794 [50] WPIX  
 DNC C2003-143576  
 TI Precipitation and retention of particle e.g. drug in carrier, by dissolving material in pressurized **gaseous** fluid or **solvent**, precipitating particles, directing into carrier mixed bed in mixed state and dispersing to produce blend.  
 DC B02 B03 B07  
 IN BOCHNIAK, D J; HORHOTA, S; KOENIG, K J; SAIM,  
 S  
 PA (BOEH) BOEHRINGER INGELHEIM PHARM INC  
 CYC 101  
 PI US 2003066800 A1 20030410 (200350)\* 37p B01D011-00 <--  
 WO 2003030871 A1 20030417 (200350) EN A61K009-16  
 RW: AT BE BG CH CY CZ DE DK EA EE ES FI FR GB GH GM GR IE IT KE LS LU  
 MC MW MZ NL OA PT SD SE SK SL SZ TR TZ UG ZM ZW  
 W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ DE DK  
 DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR  
 KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ OM PH PL PT  
 RO RU SD SE SG SI SK SL TJ TM TN TR TT TZ UA UG UZ VC VN YU ZA ZM  
 ZW  
 ADT US 2003066800 A1 Provisional US 2001-328301P 20011010, US 2002-268879  
 20021010; WO 2003030871 A1 WO 2002-US32303 20021010  
 PRAI US 2001-328301P 20011010; US 2002-268879 20021010  
 IC ICM A61K009-16; B01D011-00  
 AB US2003066800 A UPAB: 20030805  
 NOVELTY - Particle precipitation and retention in carrier material (CM), involves dissolving a solid or semisolid material (SSM) in a pressurized **gaseous** fluid or in a liquid **solvent**, precipitating particles, directing into a mixed bed of CM and retaining and dispersing the particles in CM to produce a blend of the SSM particles and CM. The CM in the mixed bed is maintained in a mixed state.  
 DETAILED DESCRIPTION - Particle precipitation and retention in carrier material (CM) comprises dissolving a solid or semisolid material (SSM) in a pressurized **gaseous** fluid or in a liquid **solvent**, to form a solution comprising a **gaseous** or liquid fluid **solvent** and a dissolved **solute** of material, precipitating SSM particles out of **gaseous** or liquid fluid solution by introducing into a region of lower pressure or into a region containing an inert **gas**, directing the introduced solution and precipitated particles onto or into a mixed bed of carrier material and retaining and dispersing the precipitated particles in the carrier material to produce a blend of the solid or semisolid material particles and carrier material, a granulation of the solid or semisolid material particles with carrier material and/or partially or fully coated with the solid or semisolid material. The carrier material in the mixed bed is maintained in a mixed state.  
 USE - Used for processing solution particles used in pharmaceuticals and chemical processing to obtain fine powders of drug substance. The method can be used in blending **crystallized** microparticles and nanoparticles with larger sized material and for coating of granules, pellets, non-pareils, tablets or capsules.  
 ADVANTAGE - The method facilitates precipitation of **solute** particle and retention and dispersion in a carrier material using pressurized **gaseous** fluids having unique properties. The method facilitates discharging and handling of the powder in downstream

processing.

Dwg.0/21

FS CPI

FA AB; DCN

MC CPI: B06-D04; B06-E05; B07-A02B; B07-D04B; B11-B; B12-M11D

TECH UPTX: 20030805

**TECHNOLOGY FOCUS - PHARMACEUTICALS - Preferred Components:** The precipitated particles of solid or semi-solid material (SSM) comprise microparticles or nanoparticles of SSMs. SSM Comprises physiologically active material, an encapsulating material, a moisture protection material, light protection material, **gas** protection material, diffusion barrier material or a dissolution or dispersion enhancing material. The active material comprises ipratropium bromide, tiotropium bromide, oxytropium bromide or tipranavir.

The powdered carrier material comprises microparticles or nanoparticles of carrier material.

**Preferred Method:** The mixed bed of carrier material is maintained in a mixed state by stirring at a rate of 20-1000 (300-1000) rpm. The method produces a blend of SSM particles with carrier material. The blend of SSM particles with carrier material comprises a (non)uniform mixture of carrier material, discrete particles of SSM and carrier material having loosely adhered SSM. The coated carrier material is produced by coating several times on coated carrier material. The **gaseous** fluid solution is introduced into a region of lower pressure. The liquid solution is introduced into a region containing a pressurized **gaseous** fluid. The liquid solution is introduced into a region into which a pressurized **gaseous** fluid is subsequently introduced. The carrier material comprises lactose. The orifice through which the **gaseous** fluid solution is introduced is located within the mixed bed when the mixed bed is at rest. The SSM of active component and binder material are dissolved in the liquid **solvent** such as methanol or ethanol.

**TECHNOLOGY FOCUS - ORGANIC CHEMISTRY - Preferred Components:** The **gaseous** fluid comprises **carbon dioxide**, nitrous oxide, trifluoromethane, ethane, ethylene, propane, sulfur hexafluoride, propylene, butane, isobutane and/or pentane. The liquid **solvent** comprises water, aliphatic alcohols, acetone, dichloromethane and/or ethyl acetate. The carrier material is in the form of powder, granulated powder, tablets capsules or caplets. The carrier material comprises a carrier, adjuvant or excipient or active material.

ABEX UPTX: 20030805

**EXAMPLE** - 5 g of drug substance was mixed with diatomaceous earth in a vessel. Supercritical **carbon dioxide** was supplied into the vessel at 80degreesC and drug substance was extracted and solubilized under 310 bar. The drug-laden effluent **carbon dioxide** stream was then **expanded** to a lower pressure through a 75 micro-m orifice nozzle in a mixing vessel containing 25 g of white powder of polystyrene divinyl benzene beads (particle size of 40-80 micro-m). The powder was mixed at 1000 rpm. The nozzle lip was set close to the top of the powder bed so that the drug substance precipitated as microparticles and nanoparticles were rapidly mixed with the powder. Mixing vessel temperature and pressure were 40-50degreesC and upto 1000 psig, respectively. Effluent **carbon dioxide** was passed through a 60 micro-m filter frit and was then vented. The treated powder had a yellowish, evenly distributed color, showing that the drug was uniformly distributed throughout the powder.

**expansion-contraction.**

DC B07  
 IN BOCHNIAK, D J; HORHOTA, S; SAIM, S  
 PA (BOEH) BOEHRINGER INGELHEIM PHARM INC; (BOCH-I) BOCHNIAK D J; (HORH-I)  
 HORHOTA S; (SAIM-I) SAIM S  
 CYC 36  
 PI WO 2001066215 A1 20010913 (200168)\* EN 48p B01D011-02 <--  
 RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE TR  
 W: AU BR CA CN CZ HU IL IN JP KR MX NZ PL RU TR US ZA  
 AU 2001034659 A 20010917 (200204) B01D011-02 <--  
 US 2001055561 A1 20011227 (200206) B01D011-00 <--  
 EP 1263516 A1 20021211 (200301) EN B01D011-02 <--  
 R: AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL PT SE TR  
 BR 2001008912 A 20021224 (200309) B01D011-02 <--  
 KR 2002077523 A 20021011 (200314) B01D011-02 <--  
 CZ 2002003273 A3 20030514 (200337) B01D011-02 <--  
 HU 2003000065 A2 20030528 (200341) B01D011-02 <--  
 CN 1411389 A 20030416 (200345) B01D011-02 <--  
 ZA 2002006943 A 20030625 (200348) 53p B01D000-00 <--  
 JP 2003525731 W 20030902 (200358) 39p B01J019-00 <--  
 MX 2002008331 A1 20030101 (200373) B01D011-02 <--  
 ADT WO 2001066215 A1 WO 2001-US3019 20010130; AU 2001034659 A AU 2001-34659  
 20010130; US 2001055561 A1 Provisional US 2000-186888P 20000303, US  
 2001-774232 20010130; EP 1263516 A1 EP 2001-906792 20010130, WO  
 2001-US3019 20010130; BR 2001008912 A BR 2001-8912 20010130, WO  
 2001-US3019 20010130; KR 2002077523 A KR 2002-711573 20020903; CZ  
 2002003273 A3 WO 2001-US3019 20010130, CZ 2002-3273 20010130; HU  
 2003000065 A2 WO 2001-US3019 20010130, HU 2003-65 20010130; CN 1411389 A  
 CN 2001-806012 20010130; ZA 2002006943 A ZA 2002-6943 20020829; JP  
 2003525731 W JP 2001-564861 20010130, WO 2001-US3019 20010130; MX  
 2002008331 A1 WO 2001-US3019 20010130, MX 2002-8331 20020827  
 FDT AU 2001034659 A Based on WO 2001066215; EP 1263516 A1 Based on WO  
 2001066215; BR 2001008912 A Based on WO 2001066215; CZ 2002003273 A3 Based  
 on WO 2001066215; HU 2003000065 A2 Based on WO 2001066215; JP 2003525731 W  
 Based on WO 2001066215; MX 2002008331 A1 Based on WO 2001066215  
 PRAI US 2000-186888P 20000303; US 2001-774232 20010130  
 IC ICM B01D000-00; B01D011-00; B01D011-02;  
 B01J019-00  
 ICS B01D009-00; B01D009-02; B09B003-00  
 AB WO 2001066215 A UPAB: 20011121  

NOVELTY - A **solute** is processed by dissolving it in a **solvent**; dissolving a **gas** in the solution; causing the solution to **expand** through a filter; causing the **gas** to be dissolved to a concentration such that the solution **expands**; retaining precipitated **solute** on a filter; reducing the pressure in the solution to expel the **gas**; and optionally adding more **solute** to the resultant solution.

DETAILED DESCRIPTION - Processing a **solute** comprises

- (a) dissolving at least a portion of the **solute** in a liquid **solvent** that has an affinity for the solubilization of the **solute**;
- (b) dissolving a **gas** in the solution;
- (c) causing the solution to **expand** through a filter that can retain unsolubilized **solute** particles;
- (d) causing the **gas** to be dissolved to a concentration such that the solution **expands** until it loses its affinity for the solubilization of the **solute** and the **solute** precipitates;
- (e) retaining precipitated **solute** on a filter which is the same as or different from the filter used in step (c);
- (f) reducing the pressure in the solution such that the **gas** is expelled, providing a resultant solution having an affinity for the solubilization of the **solute**; and

(g) optionally adding more **solute** to the resultant solution.

**USE** - For processing a **solute** for, e.g. **recrystallization** of a dissolved material from a solution, extraction of material from a composition, coating of a material on a substrate, impregnating a material into a matrix, removal of contaminants from an article, or chemical reactions (claimed).

**ADVANTAGE** - The inventive process employs minimum consumption of the organic **solvent** and **gas**, and reduced operating and capital costs. It operates at low temperatures and pressures such that environmental friendliness is enhanced. The **solvent** can easily be adjusted and can be reused for extraction, and little or no extract is typically lost.

Dwg.0/10

FS CPI

FA AB; DCN

MC CPI: B05-C04; B11-C01

TECH UPTX: 20011121

**TECHNOLOGY FOCUS - CHEMICAL ENGINEERING** - Preferred Method: Steps (a)-(f) are repeated at least one more time, or steps (a)-(g) are repeated at least three times.

**TECHNOLOGY FOCUS - PHARMACEUTICALS** - Preferred Material: The **solute** is a pharmaceutical drug substance, an impurity, or an intermediate product in the synthesis of a pharmaceutical drug substance.

**TECHNOLOGY FOCUS - INORGANIC CHEMISTRY** - Preferred Material: The **gas** is **carbon dioxide**.

L116 ANSWER 4 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN  
 AN 2000-387648 [33] WPIX  
 DNC C2000-117665  
 TI Continuous harvesting of particles from organic solution-laden near critical and supercritical fluids uses filter consisting of thin membrane supported on sintered stainless steel tube.  
 DC B07  
 IN BOCHNIAK, D J; RAJEWSKI, R A; SUBRAMANIAM, B  
 PA (UNIV) UNIV KANSAS  
 CYC 87  
 PI WO 2000029096 A1 20000525 (200033)\* EN 30p B01D061-00 <--  
 RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW NL  
 OA PT SD SE SL SZ UG ZW  
 W: AE AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB  
 GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU  
 LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR  
 TT UA UG UZ VN YU ZA ZW  
 AU 9958183 A 20000605 (200042) B01D061-00 <--  
 US 6113795 A 20000905 (200044) B01D061-00 <--  
 EP 1133345 A1 20010919 (200155) EN B01D061-00 <--  
 R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK NL PT  
 RO SE SI  
 AU 753461 B 20021017 (200280) B01D061-00 <--  
 ADT WO 2000029096 A1 WO 1999-US20651 19990909; AU 9958183 A AU 1999-58183  
 19990909; US 6113795 A US 1998-193660 19981117; EP 1133345 A1 EP  
 1999-945612 19990909, WO 1999-US20651 19990909; AU 753461 B AU 1999-58183  
 19990909  
 FDT AU 9958183 A Based on WO 2000029096; EP 1133345 A1 Based on WO 2000029096;  
 AU 753461 B Previous Publ. AU 9958183, Based on WO 2000029096  
 PRAI US 1998-193660 19981117  
 IC ICM B01D061-00  
 AB WO 200029096 A UPAB: 20000712  
 NOVELTY - A feed stream is fed into the separator, containing a porous layer (56), at a pressure of 0.5 to 1 Ps. The feed stream consists of the

particles and a mixture including a **solvent** and an **antisolvent** for the particles. The feed stream contacts the porous layer (56). At least some of the mixture passes through the layer and at least some of the particles are separated by it.

**DETAILED DESCRIPTION** - The **antisolvent** may be **carbon dioxide**, propane, butane, isobutane, nitrous oxide, sulfur hexafluoride, trifluoromethane, methane, hydrogen, or mixtures of these. The feed stream is introduced under supercritical conditions for the mixture. The **solvent** is miscible with the **antisolvent** at this pressure. The **solvent** is an organic **solvent**. The separator consists of two porous layers, the first (70) being a membrane of titanium dioxide with a thickness of 0.5 to 40 microns, and the second (72) a porous sintered stainless steel. The feed stream is prepared prior to being introduced to the separator by contacting the **antisolvent** with a dispersion including a **solute** dissolved in the **solvent** so that at least some of the **solute** precipitates out of the dispersion to form the particles.

**USE** - For continuously harvesting micro- and nano-particles from near-critical or supercritical fluids. In specific examples, the particles are pharmaceuticals, e.g. a cancer treating agent, a pharmaceutical for use in intravenous injections or particles for use in inhalation therapy.

**ADVANTAGE** - The method provides an increased rate of production and harvesting. No chemical reactions take place in the process resulting in particles which are the same chemically as the drug used to form the dispersion.

**DESCRIPTION OF DRAWING(S)** - The figure shows schematically the high pressure filter.

porous layer 56

porous membrane 70

sintered stainless steel tube 72

Dwg. 2/6

FS CPI

FA AB; GI; DCN

MC CPI: B05-C03; B05-C07; B05-C08; B10-H02B; B10-J02; B11-B; B12-M11E;  
B14-H01

L116 ANSWER 5 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 2000-183881 [17] WPIX

DNC C2000-057847

TI Production of spherical particles, especially of e.g. pharmaceuticals, comprises **crystallization** on spherical seed **crystals**.

DC B04

IN HEFFELS, S; NICOLAOU, I; SCHUNK, W

PA (AVET) AVENTIS RES & TECHNOLOGIES GMBH & CO KG; (AXIV-N) AXIVA GMBH

CYC 25

PI DE 19834876 A1 20000203 (200017)\* 6p B01D009-02 <--  
WO 2000007685 A1 20000217 (200017) DE B01D009-00 <--  
RW: AT BE CH CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE  
W: BR BY IN MX PL RU US

ADT DE 19834876 A1 DE 1998-19834876 19980801; WO 2000007685 A1 WO 1999-EP4787  
19990708

PRAI DE 1998-19834876 19980801

IC ICM B01D009-00; B01D009-02

ICS A61K009-10; A61K009-16

AB DE 19834876 A UPAB: 20000405

**NOVELTY** - Process (A) for producing particles comprises **crystallization** using spherical seed **crystals**.

**DETAILED DESCRIPTION** - INDEPENDENT CLAIMS are also included for the following:

(1) a process (B) for producing spherical seed **crystals**, comprising spray drying a solution of the substance to be **crystallized**;

(2) a process (C) for producing spherical particles, comprising dispersing the substance to be **crystallized** in an immiscible organic **solvent** and **crystallizing** the substance in the resulting droplets;

(3) particles obtainable by processes (A), (B) or (C).

USE - The process is especially useful for producing spherical particles of pharmaceuticals, e.g. cefotaxime disodium or piratenide, or special chemicals, e.g. phenylhydrazines, either by **crystallization** from melts, solutions, **gases** or supercritical media or by precipitation or reactive **crystallization**, optionally where the particles comprise several shell-like layers, at least two of which have a different composition.

ADVANTAGE - Spherical **crystals**, which have good flow properties, can be produced without the need for large-scale extractive **crystallization** in droplets dispersed in an immiscible **solvent**. The spherical seed **crystals** can be produced by simple spray drying.

Dwg.0/3

FS CPI

FA AB; DCN

MC CPI: B06-F03; B10-A19; B11-B

ABEX UPTX: 20000405

EXAMPLE - A 15% aqueous solution of cefodizime disodium (I) was spray dried with nitrogen to produce spherical particles with a size of 9 mum. An aqueous solution of (I) was **crystallized** by dilution with ethanol in the presence of 1 weight% (based on **solute**) of the spherical particles. The product was filtered and dried to give largely spherical **crystals** with an average particle size of 16 mum.

L116 ANSWER 6 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 1994-349384 [43] WPIX

DNC C1994-159105

TI Gas-evolution separation of a **solute** from solution in a **solvent** - by dissolving a **gas** forming material in the soln and adjusting pressure and temperature to provide a three phase separation.

DC D15 E33 J01 M25

IN FERRAMOSCA, A C; LURIE, W; SLOAN, J C

PA (PARH-N) PARHELION INC

CYC 1

PI US 5360554 A 19941101 (199443)\* EN 17p C02F001-22

ADT US 5360554 A US 1994-192725 19940207

PRAI US 1994-192725 19940207

IC ICM C02F001-22

ICS B01D009-04

AB US 5360554 A UPAB: 19941216

**Solute** is separated from a solution in a **solvent** by dissolving a **gas** forming material (fr.40) in the solution (16) lowering the temperature of the solution placing the solution in a pressure vessel (52) and increasing its pressure to a nominal high value from which it is released to allow the major portion of the **gas** forming material and a minor portion of the **solvent** to form vapours that undergo a Joule-Thompson free **expansion** into a closed second vessel (40) at a low pressure to obtain three phases of resultant materials that each have a temperature approximating the triple point temperature of the solution. The three phases comprise a **gas** phase product containing the **gas** forming material and vapours of the **solvent**, a liquid phase product with **solute** concentration greater than the initial concentrate of the **solute**/**solvent** soln starting material, and a solid phase form of the **solvent**. One of the phases is collected as product of the process, a portion of the **gas** phase product is collected from reuse (at 26) in the process, a portion of the **gas** phase product is

collected from reuse (at 26) in the process, and one of the phases is recycled into an earlier stage of the process via a heat exchanger (38) that heat exchanges a relatively cold resultant material with a relatively warm **solute/solvent** solution. Also claimed is the process in which the liquid phase product with increased **solute** concentration is recycled (86,96) to the initial **solute/solvent** soln to adjust its **solute** concentration. Heat exchangers (46,48) are used to control the temperature of the solution prior to pressurising in the pressure vessel, a jet eductor (24) is used to collect the **gas** phase product for re-use, and the liquid product is either recycled via a heat exchanger, or separated into a constant recycle quantity and a remaining "Blow-Down" quantity comprising total **solute** of the starting material and unsolidified remaining **solvent** in proportion up to the eutectic proportion of the starting **solute/solvent** solution.

**USE** - In converting sea or brackish water into potable water, recovering metals such as magnesium from sea water etc. de mineralising fresh water to make carbonated beverages, recovering **solutes** or **solvents** from industrial process **solvents**, or cleaning up polluted bodies of water.

**ADVANTAGE** - Only the vapour products are **expanded** into the second vessel reducing energy expenditure yet producing substantial quantities of solid **solvent**.

Dwg.2/2

FS CPI  
FA AB; GI; DCN  
MC CPI: D04-A01F; E11-Q01; E31-N05C; E34-B; J01-C; J01-D; M25-F; M25-G16

L116 ANSWER 7 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN  
 AN 1991-261186 [36] WPIX  
 CR 1991-347857 [48]; 1993-183877 [23]; 1993-338219 [43]  
 DNC C1991-113366  
 TI Supercritical fluid extraction with independent control of conditions - comprises which system, maintains **solvent**, flow, temperature and pressure using a variable **expansion** nozzle.  
 DC J01  
 IN DRYDEN, P C; ENGEL, S J; FRANK, L R; WURM, C M  
 PA (HEWP) HEWLETT-PACKARD CO  
 CYC 5  
 PI EP 444299 A 19910904 (199136)\*  
 R: DE FR GB  
 US 5133859 A 19920728 (199233) 13p B01D015-08 <--  
 JP 04222602 A 19920812 (199239) 11p B01D011-00 <--  
 EP 444299 B1 19950222 (199512) EN 15p B01D011-02 <--  
 R: DE FR GB  
 DE 69017190 E 19950330 (199518) B01D011-02 <--  
 JP 3207866 B2 20010910 (200155) 10p B01D011-00 <--  
 ADT EP 444299 A EP 1990-125072 19901221; US 5133859 A US 1990-487693 19900302;  
 JP 04222602 A JP 1991-59543 19910301; EP 444299 B1 EP 1990-125072  
 19901221; DE 69017190 E DE 1990-617190 19901221, EP 1990-125072 19901221;  
 JP 3207866 B2 JP 1991-59543 19910301  
 FDT DE 69017190 E Based on EP 444299; JP 3207866 B2 Previous Publ. JP 04222602  
 PRAI US 1990-487693 19900302  
 REP EP 206739; EP 275933; EP 296145; JP 06214885; JP 62148855  
 IC ICM B01D011-00; B01D011-02; B01D015-08  
 ICS G01N001-10  
 AB EP 444299 A UPAB: 20010927  
 A method for the supercritical extraction of one component of a sample uses a flow system having control equipment for the pressure and temperature of the extraction medium. The control equipment operates in conjunction with a variable flow restrictor nozzle to control the condition of the fluid flowing through the sample chamber (. Pressurised extraction fluid is supplied from a cylinder via a pump ( with a pressure regulator to the

sample chamber.

The fluid is exhausted via the **expansion nozzle** (. The pump injects the fluid into the system at a controlled, predetermined, flowrate and the system pressure is controlled by setting the variable nozzle as appropriate. The equipment also controls to a predetermined extraction time.

Pref. additional features include a bypass to allow the **solvent** to be routed away from the sample chamber, and a nozzle and trap system for collecting a sample of the material after the extraction. Rinse **solvent** may be passed through the sample trap after collection to remove selected fractions for analysis or further treatment. A sample of **solvent** containing the extracted **solute** may also be collected after it has left the extraction chamber.

**USE/ADVANTAGE** - Improved method of supercritical fluid extraction. Control system allows independent selection and control of pressure and temperature of extraction medium (i.e., its **solvent** power). Appts. can use any convenient vessel as a sample container. @ (12pp Dwg. No. 0/3)

FS CPI

FA AB

MC CPI: J01-C01

ABEQ US 5133859 A UPAB: 19930928

Appts. for components extn. from a sample, specifically by **gas** or liq. chromatography or supercritical fluid chromatography, comprises a **gas** liquefaction pump controlled to have regulated output pressure for supplying a chamber contg. the sample via a variable orifice nozzle controlled by a pressure transducer, so that the **gas** pressure in the chamber is at a set point value.

Pref. the **gas** exits the chamber via a trap contg. porous granular material, which is inert, chemically active or adsorbent.

**USE** - Chromatography using liq. CO<sub>2</sub> as sample component extn. **solvent**.

ABEQ EP 444299 B UPAB: 19950328

Apparatus for the extraction of components from a sample comprising: (a) one or more sources of **solvent** fluid (100); (b) one or more extraction **solvent** fluid input ports (101), (c) a controllable high pressure pump (202); (d) a pressure transducer (240a) to measure the pressure of the fluid delivered by the high pressure pump (202); (e) a flow transducer (226); (f) an **expansion** nozzle section having a variable and controllable flow restriction (108); (g) a control apparatus for controlling independently the variable flow restriction and the high pressure pump (202) so as to achieve and to maintain a set point pressure and a set point flow rate; (h) an extraction chamber flow system (209) comprising an extraction chamber (210) for retaining the sample in the flow stream of the fluid and a sample input module for containing the sample in the extraction chamber (210); (i) a bypass flow system (207) which routes fluid flow around said extraction chamber section (k) means (213) for merging the bypass flow system (207) and extraction chamber flow system (209) together; (l) sample collection means for separating the extracting **solvent** fluid from components from said sample, comprising at least one nozzle (216) and trap (218) subassembly, said nozzle forming part of said **expansion** nozzle section; and (m) at least one sample collection vessel (236) for collecting the components from said sample.

Dwg. 3/3

L116 ANSWER 8 OF 8 WPIX COPYRIGHT 2004 THOMSON DERWENT on STN

AN 1981-28359D [16] WPIX

TI Purificn. of crystalline solid from solution - by pressurising, releasing pressure and discharging **solvent** by pressure of **gas** (J5 19.8.75).

DC J01

PA (KOBM) KOBE STEEL LTD

CYC 1

PI JP 56012161 B 19810319 (198116)\*  
JP 50104771 A 19750819 (198116)

PRAI JP 1974-11328 19740125

IC B01D009-02

AB JP 81012161 B UPAB: 19930915

Method comprises pressurising solution in a pressure vessel to solidify the solute, releasing the pressure rapidly to such a level as to re-dissolve a part of the solid, and discharging the solvent by pressure of gas which was initially charged in the vessel.

Used for purifying **crystalline** solid from solution  
(J50104771).

FS CPI

FA AB

MC CPI: J01-B

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